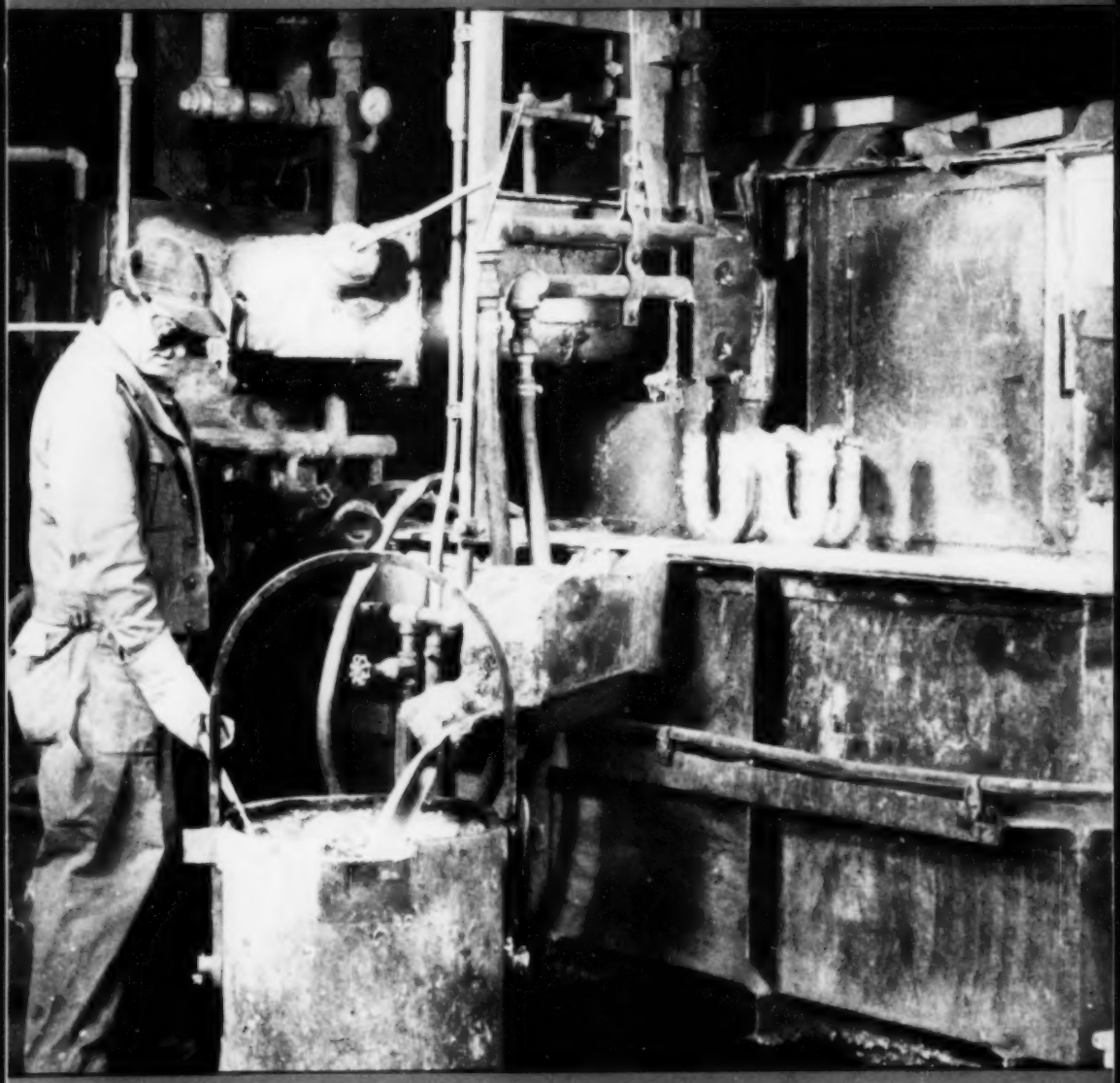
JUNE, 1952
IN TWO SECTIONS—SECTION I

Journal of METALS



June JOURNAL OF METALS Highlights:

"The Sterling Process" - p. 581
"Foreign Ores as Open Hearth Charge Ore" - p. 592
"Effect of Zr on Mg-Th and Mg-Th-Ce Alloys" - p. 633

Instrument panel at discharge end of No. 11 rotary kiln at Maple Grove. Automatic control and recording of firing temperatures play an important role in protecting product quality.

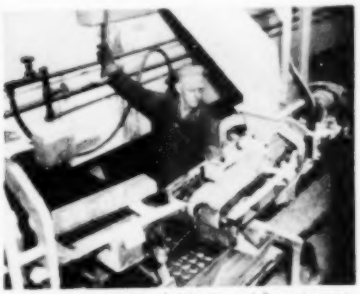
Two new rotary kilns, shown in process of erection at right above, will soon add materially to dead burned dolomite production at the Maple Grove plant.

Done to a turn

Quality in dead burned dolomite is proved in the open hearth furnace. It is measured by refractory cost per ton of steel.

Quality in Basic refractories is established and guarded by materials, machines and men—by expert processing of high grade ores, scientific proportioning and blending of these ores with fluxes, closely controlled burning, careful inspecting and laboratory testing at every stage of production.

No wonder Basic products prove so uniformly dependable, help steel makers reduce refractory costs



Accurate proportioning of dolomite and fluxes is essential to quality. Here, additives (entering on left) are blended with dolomite, which is traveling via conveyor to the kiln.

Test furnaces in the Basic Research Laboratory at Bettsville, Ohio, keep busy with quality control of refractory production and development of new products.

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Journal of **METALS**

VOL. 4, NO. 6 — JUNE, 1952

In Two Sections — Section 1

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COVER

The exclusive story of New Jersey Zinc's Sterling Process, a new method for smelting zinc ores in an electric furnace, begins on page 581 of this issue. The cover shows molten zinc being tapped from the condenser reservoir in one of the steps of the process.

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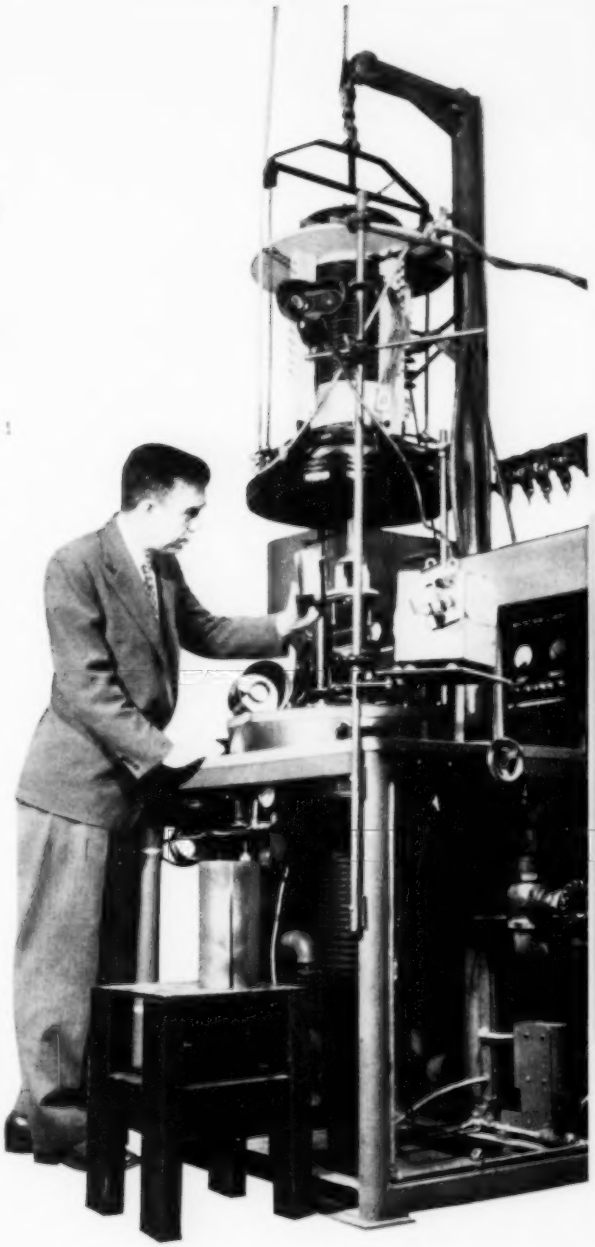

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ELECTROMET *Data Sheet*

A Digest of the Production, Properties, and Uses of Steels and Other Metals

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Extra-Low-Carbon STAINLESS STEEL

New Type Chromium-Nickel Steels Have Added Corrosion Resistance

New and improved austenitic stainless steels of the 18-8 type have been developed which have superior corrosion resistance after being exposed to heat. These steels, known as extra-low-carbon stainless steels, were designed especially for use in welded and stress-relieved equipment that is exposed to more severe corrosive conditions than are normally encountered by other types of straight 18-8 stainless steel.

Under severe corrosive conditions, intergranular attack may occur in some of the higher carbon grades of austenitic stainless steels that have been subjected to the temperature range of 800 to 1600 deg. F. during welding or hot forming operations. It is generally agreed that this type of corrosion is caused by complex carbides that are formed at the grain boundaries of the stainless steel during heating.

The effect of heat is rarely harmful in the ordinary fabrication of stainless steel for most applications, such as in architecture, the food and dairy industries, in hospitals, and in the home. However, in the chemical and other allied industries, where stainless steel is used in the handling of very corrosive chemicals, these new extra-low-carbon stainless steels should most certainly find wide use.

In general, there are three ways in which the precipitation of carbides can be controlled in stainless steel:

1. Heat treating so that the carbides present are dissolved.
2. Alloying with an element, such as columbium, tantalum, or titanium, that will tie up the carbon in the form of a harmless carbide.
3. Decreasing the carbon content of the steel.

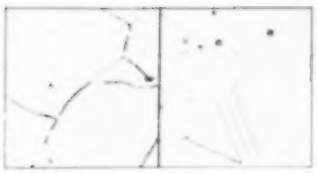

Fig. 1 Left: Carbide precipitation at the grain boundaries of an 18-8 stainless steel containing 0.09 per cent carbon after being held at 1200 deg. F. for 1 hour. Right: Absence of carbide precipitation in 18-8 stainless steel of 0.03 maximum carbon content after being held at 1200 deg. F. for the same length of time.

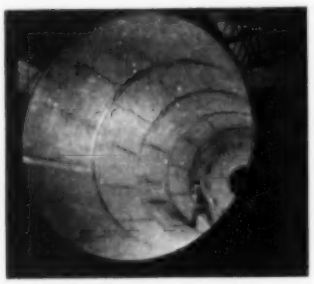

Fig. 2 The new extra-low-carbon stainless steels are especially suited for large types of process equipment, such as this fractionating tower. They require no heat treatment after welding.

Heat-Treatment After Welding

Before the development of extra-low-carbon stainless steel, or of the "stabilized grades," one means for preventing intergranular corrosion was to heat treat stainless steel that had been subjected to the dangerous temperature range, so that the precipitated chromium carbides would go back into solid solution. It was found that when a welded part was heated to temperatures of 1950 to 2000 deg. F., and then cooled rapidly, most of the carbides were retained in solid solution. This extra heat treatment is sometimes impractical, however, because of the design or massive size of some types of welded equipment.

Decreasing Carbon Content

A recent development in preventing intergranular corrosion has been the extra-low-carbon stainless steels. To be substantially harmless in stainless steel for as-welded or welded and stress relieved chemical equipment operating at temperatures under 700 deg. F., carbon must not be present in quantities over 0.03 per cent.

In 1947, ferrochrome with 0.03 per cent maximum carbon was first produced for the steel industry by ELECTROMET. This product has helped make it possible to produce very low-carbon stainless steels—steels that are completely immune to intergranular corrosion after welding or after subjection to a stress-relieving heat treatment.

The amount of stabilizing element that is necessary to "fix" carbon in stainless steel is in direct proportion to the carbon content of the steel. Therefore, the lower the carbon the less is the amount of stabilizing element required. Lowering the carbon content is an efficient means of conserving columbium, tantalum, and titanium.

Metallurgical Service Available

If you use welded stainless steel equipment, it will pay you to investigate the advantages of using extra-low-carbon steels. If you produce stainless steel, our metallurgists will be glad to give you technical assistance in the use of ferrochrome of 0.03 per cent maximum carbon. For further information, write to the nearest ELECTROMET office.

For a more detailed account of the properties of extra-low-carbon stainless steel, write for a free copy of the technical paper, "Resistance to Sensitization of Austenitic Chromium-Nickel Steels of 0.03% Max. Carbon Content".

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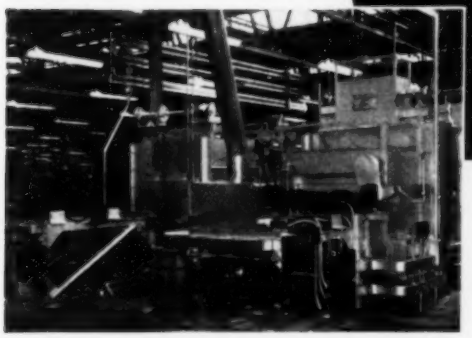

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MEET THE AUTHORS

Bolesław L. Dunicz (p. 604) received his M.Sc. in 1935 from the King John Casimir University in Lwów, Poland. Prior to World War II, he was on the staff of the University teaching analytical chemistry. During the war, Dr. Dunicz was in England and obtained his Ph.D. at King's College, University of London. Presently engaged in research at the Institute for the Study of Metals, Chicago, he counts swimming and playing the violin as his hobbies. He is also interested in the arts and music. Dr. Dunicz is also a member of the American Chemical Society and American Assn. for the Advancement of Science.

B. L. DUNICZ

D. S. EPPELSHEIMER

D. S. Eppelsheimer (p. 615), professor of metallurgical engineering at the Missouri School of Mines and Metallurgy, Rolla, is an alumnus of Harvard. In 1933, Dr. Eppelsheimer was research assistant at Harvard and then joined Union Carbide & Carbon Research Laboratories at Niagara Falls. From 1938 to 1945 he was professor of industrial engineering, experiment station, University of New Hampshire. He was also associated with Metal Hydrides, Inc., Beverly, Mass. A member of the St. Louis AIME section, he is also a member of numerous professional societies both here and abroad. His spare time is spent collecting stamps and antique guns, and farming.

H. G. Blocker (p. 657), graduate of Georgia Institute of Technology (B.M.E.), was born in Tampa. In 1942 Mr. Blocker was a machinist for the General Auto Supply Co. After a hitch in the Navy during the war, he returned to college, and is presently a research assistant at the state engineering experiment station, Atlanta. In his leisure hours, Mr. Blocker enjoys music and camping.

H. C. Chang (p. 639) was born in Hopei, China. A graduate of Wu han University (B.S.) and Case Institute of Technology (M.S.), he is now at Massachusetts Institute of Technology working for his D.Sc. From 1942 to 1947 he worked in electrochemical and metallurgical plants, Szechuan, China. Photography occupies Mr. Chang in his free moments.

H. C. CHANG

Susan L. Craig (p. 657) is a research assistant at the state engineering experiment station at the Georgia Institute of Technology. An alumna of Florence State Teachers (B.C.) Mrs. Craig was a teacher in Leighton, Ala. In 1949 she joined the Reynolds Metals Co. as a chemist at Listerhill. In January she became associated with the Georgia Institute of Technology. After her wifely chores are finished, Mrs. Craig likes fishing.

J. B. Hess (p. 645), research metallurgist, Kaiser Aluminum & Chemical Corp., Spokane, was born at Bloomsburg, Pa. After college graduation (Massachusetts Institute of Technology, S.B.) he joined J. H. Williams & Co. In 1947 Mr. Hess was research metallurgist in the magnesium laboratories of the Dow Chemical Co. For five years he was at the Institute for the Study of Metals at University of Chicago and in 1951 joined Kaiser. This AIME member has presented two other papers. He is a member of ASM and the British Institute of Metals.

J. B. HESS

Earl S. Greiner (p. 646) has been a member of the technical staff at the Bell Telephone Laboratories, Murray Hill, N. J. since 1930. A graduate of Carnegie Institute of Technology (B.S.), Lehigh University (M.S.), and Columbia University (Ph.D.), he enjoys photography as a hobby.

Terkel Rosenqvist (p. 604) is assistant professor and lecturer in the dept. of metallurgy, Norway's Institute of Technology, Trondheim. Born in Oslo, Dr. Rosenqvist attended the University of Oslo. He was employed by various Norwegian metallurgical plants, and from 1947 to 1951 was assistant professor at the Institute for the Study of Metals. He has authored several AIME papers and is a member of various professional societies. In this country of winter sports, skiing and hiking are enjoyed by Dr. Rosenqvist.

C. Orr, Jr. (p. 657), graduate of University of Tennessee (B.S. and M.S. chemical engineering) was born at Lewisburg, Tenn. After discharge from the service in 1946 Mr. Orr was employed as a chemical engineer by the Tennessee Valley Authority. He has been engaged in research and graduate study at Georgia Institute of Technology and since July 1951 has been an assistant professor.

C. ORR, JR.

H. E. Stauss (p. 656) is head of the electric and magnetic alloys branch, Naval Research Laboratory, Washington, D. C. An alumnus of Washington University and University of California, he holds an A.B., M.A., and Ph.D. Dr. Stauss had been an instructor of physics at the University of Nebraska from 1927 to 1929. He also served as physicist and metallurgist for Baker & Co., Inc., Newark, N. J. In 1942 he joined the War Production Board as chief of the precious metals and mercury section. Since 1945 he has been with the Naval Research Laboratory. Now residing in Fairfax county, Va., this AIME member enjoys gardening. He has also authored several previous papers.

D. N. Williams (p. 615), research fellow at the Missouri School of Mines and Metallurgy, was born at Salt Lake City. He attended Michigan College of Mining and Technology (B.S., M.S.) and is presently working for his Ph.D. Mr. Williams counts fishing and photography as hobbies.

D. N. WILLIAMS

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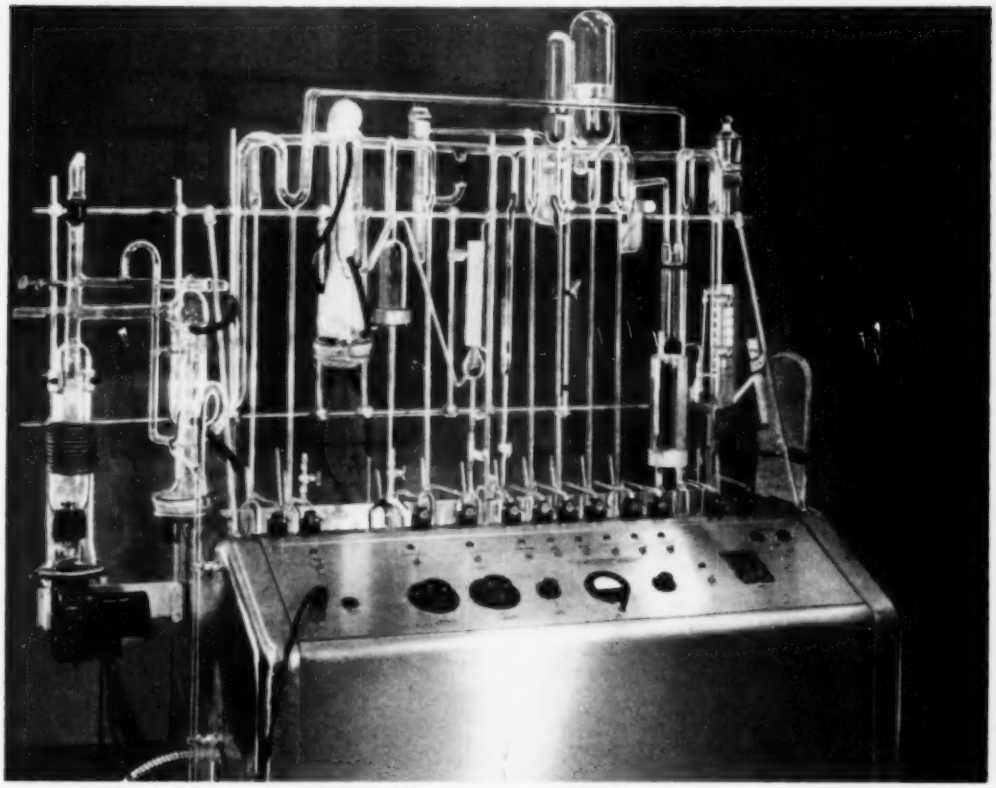

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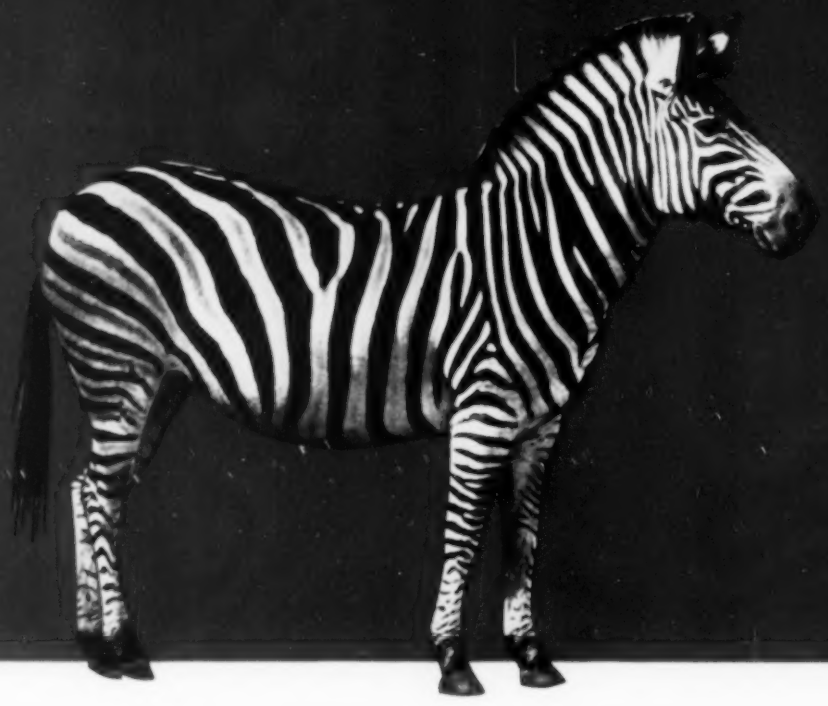

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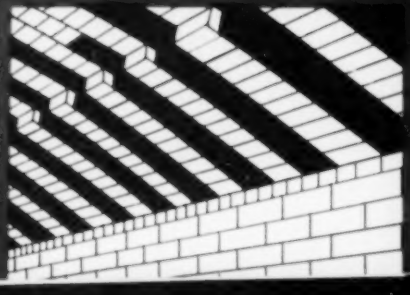

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Metallurgist, recent graduate preferred, who is interested in gaining experience in the manufacture of steel, wire and wire products. Location, northern New Jersey. Y6628

Mining or Metallurgical Engineer, with degree in mineral dressing engineering, not over 28, preferably with one or two years' experience in the milling of metals for sales contact work. Prefer single man. Salary, $4800 to $5400 a year. Location, New York. Y6494

Research Engineer, 25 to 40, graduate in mining or metallurgical engineering, preferably with a Master's degree in mineral dressing, with at least three years' experience in ore dressing, research or mill work, preferably with experience in nonmetallic flotation. Location, Florida. Y6465

Control Metallurgist with some experience in induction melting of stainless and high temperature alloys, to be responsible for incoming and outgoing materials, inspection and pouring. Location, Ohio. Y6463

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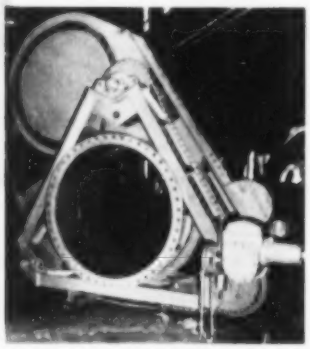
1—BORING MILL: New 3-in. horizontal boring mill announced by *Diamond Machine Tool Co.* has 18 feeds with rapid traverse in all directions, and heavy 10-hp motor with amperage load regulator. Approximate weight is 10,120 lb; spindle diam is 3 in.; platform diam is 25 in.; and dimensions of the turntable are 34½x25¼ in.

2—ARC WELDED DIAPHRAGMS: Diaphragms in thicknesses from 0.0015 to 0.005 in. for pressure elements and seals for instrument and aircraft industries are being arc welded by *Bristol Co.*

3—MAGNETIC PROBE: Made of super magnetic alloy, *General Service Equipment Co.* product is no larger than a fountain pen. It is particularly useful for removing steel or iron particles from operating areas of electrical instruments or delicate mechanisms. The strength of the magnet can be controlled by amount of extension.

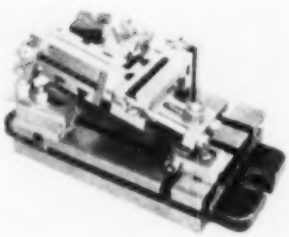
4—GOGGLE PLATE VALVE: Electrical operation and push button control is a new feature added to the *Salem-Brosius, Inc.* goggle type plate valves for large diameter blast



furnace and coke oven gas mains. Two heavy duty electrical units have been incorporated into the design that control and operate both the plate swing in the valve and the opening and closing of the valve proper. Easy operation is assured and also positive positioning of the valve in all sizes ranging from 36 to 100 in. diam.

5—MASONRY PAINT: A one-coat masonry paint has been introduced by *United Laboratories, Inc.* It is applicable to interior and exterior surfaces of concrete block, asbestos cement, and brick. It may be applied by brush or spray.

6—JIG AND FIXTURE: An additional aid to existing equipment, this universal jig and fixture system has unlimited applications for development and small quantity



production. Jigs and fixtures can be erected in a short time and after serving their purpose dismantled, the elements then being available for further erections. The underlying principle governing the design of these elements is that the interlocking of the elements, one with another, to maintain alignment with rigidity within the structure is obtained by tensioning. *Wharton & Willcocks of America, Inc.*

7—METAL CUTTING MACHINES: Machine capable of heavy foundry operations such as cutting risers up to 4 in. diam on iron castings and risers up to 2 in. diam for nickel and steel castings has been announced by *DeWalt, Inc.* A 10-hp metal cutter specializes in cutting billets, heavy tubing or various sections for cutoff operations.

8—CONVEYOR SYSTEM: Use of 9/16-in. steel core cable in joining trolley brackets was accomplished by *Dingle-Gaboury, Inc.* With the use of a new bracket assembly pull loads up to 10,500 lb are possible without indications of slipping.

9—BLAST CLEANER: A rotating table blast cleaner unit that speeds casting cleaning is available from *Pangborn Corp.* The cleaning table consists of a 9-ft diam rotating table carrying six 36-in. tables that rotate about their own axes. Abrasives bombard the work in the enclosed blasting chamber from a 45° angle. A dust control unit draws dust from the unit before the particles enter the atmosphere.

10—ATOMIC COMPOUND: An anti-radioactivity liquid compound used for removing radioactive contamination from laboratory equipment is offered by *Atomlab, Inc.* for popular application. It cleanses surfaces free from corrosion and oxidation products which tarnish aluminum, brass, copper, iron, and other metals.

11—CHIPS SEPARATOR: A new separator for effective removal of parts from deburring and finishing chips, and for grading different sizes of chips is now offered by *Gravi-o-Flo Corp.* Area occupied by the machine is 3x6 ft. with pans only 3x8 ft.

12—JOLT ROLLOVER MACHINE: After rollover it is unnecessary to raise a separate draw cylinder and equalize with this new *Johnston & Jennings* machine. Also eliminated is the unclamping operation necessary in standard rollover cycle.

13—POWDER CAMERAS: X-ray diffraction powder cameras have been announced by *North American Philips Co., Inc.* These cameras will record up to 17A spacings with cop-

per radiation, with low air scatter and unusually clear patterns in the forward reflection region.

14—SAND CONTROLLER: An automatic sand testing unit that determines and records permeability, green strength, green deformation and temperature of molding sand, has been introduced by *Harry W. Dietert Co.* It is entirely automatic and performs without manual manipulations. The controller is 33x21 in. and is 55 in. in height.

15—PUSHBUTTON UNITS: Advanced design features have been in-

corporated in a new line of oil tight pushbutton units for machine tool applications announced by *General Electric Co.* Offered with 1, 2, 3, 4, 6, and 9 button stations the lightweight, aluminum enclosures are fully oil tight and provide maximum dust protection. Contacts are rated at 115, 230, 460, and 575 v.

16—RUST INHIBITOR: This product has been formulated to give rust protection to precision parts. It can be applied to a wet, damp, or moist surface and is nontoxic and noninflammable. *Crown Industrial Products Co.*

Free Literature

21—GRINDING WHEELS: Specific wheels for cutting off, precision grinding, and snagging the billets are fully described in manual 645 issued by *Electro Refractories & Abrasives Corp.*

22—AIR VALVE: Catalog published by *Rivett Lathe & Grinder, Inc.* offers a complete file on air valves. Description of each particular model valve, working drawings, specifications, and cut-away views are included.

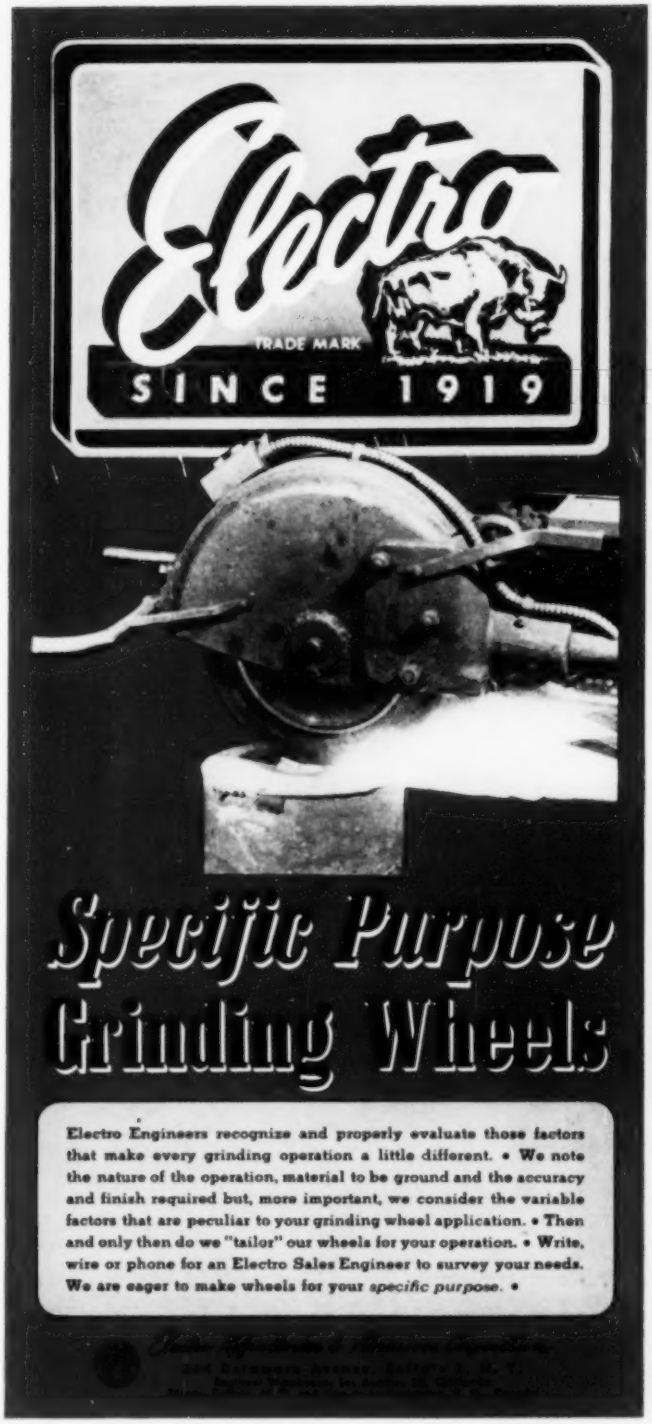
23—FOUNDRY CERAMICS: Strainer cores, gate tubes, cut-off cores, troughs and other ceramic parts are illustrated in brochure issued by *American Lava Corp.* These foundry ceramics withstand heat shock requirements.

24—FURNACES: Booklet *Proven Heat-Treating Efficiency by Loftus* is available from *Loftus Engineering Corp.* and shows many furnace types. Exclusive feature such as pin point quench assures absolutely accurate quench temperature.

25—LUBRICATION: New bulletin is obtainable from *Henry G. Thompson & Son Co.* that explains the new developed atom-lube system. It is a new atomized system designed to aid cooling and lubricating of cutting tools.

26—CARBOLOY THERMISTORS: Catalog describing Carboloy thermistors has been issued by *General Electric Co.* General basic information relating to physical and operation characteristics, applications, and wiring diagrams are included. Typical applications of standard Carboloy rod, disc, and washer-type Thermistors also are given.

27—TEMPILSTIKS®: Instruction sheet supplied by *Tempil Corp.* discusses procedures and techniques for determining temperatures in unusual applications. These temperature-sensitive crayons have calibrated melting points. Sixty differ-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ent Templitiks" cover the range from 113° to 2000 F, indicating a specified temperature within a tolerance of ± 3 per of its rating.

28—WELDING ROD COATINGS: Guide to raw materials *Foote Mineral Co.* offers manufacturers of coated welding rods is available in book form. A chart of chemical specifications of welding rod coating materials is included as well as chemical analysis.

29—CUT OFF MACHINES: Folder describes complete line of *Buehler Ltd.* cut-off machines. Five models are available for samples from ½ to 4 in. diam.

30—CAST BRONZES: Catalog on Asarco continuous cast bronzes is available from *American Smelting & Refining Co.* Physical properties, photomicrographs, table of stock shapes and sizes, and weights are contained with other valuable information.

31—INDUCTION MELTING: Bulletin *The Case for Tocco Induction Melting* is offered by *Ohio Crankshaft Co.* Rapid melting, low alloy loss, and no carbon pickup are just a few of the advantages of these furnaces for precision casting.

32—SALT BATHS: Book on liquid salt baths is obtainable from *E. F. Houghton & Co.* It describes the properties of Houghton salts and how salts eliminate scale, reduce cracking, and lower production costs.

33—IRON POWDERS: A comprehensive book on carbonyl iron powders is available. Diagrams, photomicrographs, performance charts and tables are included. Established applications are covered and new applications suggested. *Antara Chemicals Div., General Dyestuff Corp.*

34—LIQUID HELIUM CRYOSTAT: Details of the Collins helium cryostat are given in booklet JM-2 issued by *Arthur D. Little, Inc.* Superconductivity, resistance minima in metals at low temperature, and thermal conductivity are a few of the studies utilizing this Cryostat.

35—CERIUM METAL: For improving physical properties, the use of cerium metal (mischmetal) is of specific interest to the cast and wrought steel industry. Technical bulletin gives latest results of this useful alloy, a combination of cerium, lanthanum and various other rare earth elements. *General Cerium Co.*

36—NONMETALLIC SMELTING: Booklet available from *Pittsburgh Lectromelt Corp.* gives information on smelting problems. Electrical equipment and substations are built to withstand overloads.

37—HYDRIDES: Preliminary review of uses and potentials of hydrides is given in booklet obtainable from *Metal Hydrides, Inc.* The Hydrimet process is explained and various hydrides such as titanium, zirconium, sodium, and others are discussed.

38—ELECTRON MICROSCOPE: Illustrated booklet describes in detail several case histories involving the use of these electron microscopes. Typical applications by industries in chemical processing, metal fabrication, and electric fields are emphasized. *RCA Victor Div., RCA.*

39—DIE STEEL: A higher alloy steel that can be water hardened the same as straight carbon or carbon vanadium steels for cold heading dies is explained in data sheet published by *Allegheny Ludlum Steel Corp.* Certified laboratory data on analysis, proper methods of handling and shop treatment, and complete physical properties are included.

40—LOW ALLOY STEEL RUSTING: Results from exposure tests on 71 low alloy structural steels exposed in industrial atmospheres lead to new theory of the mechanism of rusting. This theory is described in booklet available from *International Nickel Co., Inc.*

41—DRILL PRESS: Bulletin issued by *Bellows Co.* shows installation photographs, describes in detail all parts of the Bellows drill press feed, and includes dimensional drawings.

42—UNIT STEAM GENERATOR: Especially prepared for engineers to understand the advance design features of the *Preferred Utilities Mfg. Corp.* generator, 28-page bulletin has been issued. Comprehensive data and dimensions, and a list of accessory equipment are included.

43—METALLOGRAPHY EQUIPMENT: Bulletin listing test specimens of common metals and alloys, mounted and identified, and supplied with photomicrograph, analysis, hardness, etc. for comparison or teaching is available from *A. J. Buehler.*

44—TEMPERING BATHS: A new bulletin on carburizing and tempering baths has been announced by *A. F. Holden Co.* Charts included show the relative case penetrations at various times and heats, amount of salt to be added each 8-hr period to balance bath and maintain operating efficiency, and instructions for controlling distortion to less than 0.001 in.

45—PRECISION CUTTING TOOLS: Carbon steel taps, prong dies, small reamers, hollow mills and centering tools, small counterborers and step drills are illustrated in catalog published by *Woodruff & Stokes Co., Inc.* Case histories are included describing how users have been able to speed production and lower costs. Information on various sizes and shapes of W-S small precision tools is also given.

46—METALLOGRAPH: Desk type metallograph is described in booklet issued by *American Optical Co.* Examining specimen through microscope automatically focuses for photography.

47—METAL CLEANERS: Bulletin describing recommended uses of each *E. F. Houghton & Co.*, alkaline, emulsion, acid phosphate and surface active agent type of cleaner is now available. Concentrations for various cleaning applications and a list of cleaners for general industrial use is included.

48—PERLITE AGGREGATE: Booklet PA-26 gives information on Permalite, perlite aggregate, obtainable from *Great Lakes Carbon Corp.*

Journal of Metals
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June

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49—SOLAR FLUX: Booklet outlining cost cutting solutions to common problems in metallic arc, inert arc, and other fusion welding of alloy steels is available from *Solar Aircraft Co.*

50—PROTECTIVE COATINGS: Entire line of *Protecoat Laboratories, Inc.* products is covered in general catalog just issued. A list of the products and brief description of physical and protective qualities is included.

51—TOOL STEELS: Tool steels designed for specific uses in machining and die work throughout the metal-working field are described in booklet published by *Universal-Cyclops Steel Corp.* Molybdenum steel, 5 pct chrome die steel and high speed vanadium steel, are a few of the various types discussed.

52—DIE FORMED RINGS: New plastic semimetallic die formed rings make it possible for manufacturers to be less demanding in the way of machined tolerances. Catalog sheet gives complete information. *Abbott & Boddie.*

53—SMOKE CONTROL: Ways and means of guarding against damage from smoke accidentally introduced in the air intake of air conditioning systems is the subject of bulletin issued by *Ess Instrument Co.*

54—FURNACES: Pit type furnaces with a work space of 48x156 in. are typical of furnaces designed and built by *Hevi Duty Electric Co.* Hevi Duty return bend type heating elements are zoned to provide uniform temperatures in the entire depth of the work chamber. Bulletin HD-1245 gives more information.

55—PARTING AGENTS: Mold release emulsion No. 35, Dow Corning 7 compound, and Dow Corning 7 emulsion are described in data sheet available from *Dow Corning Corp.* Data sheet represents conclusions drawn from laboratory testing and evaluation, as well as on the job experience.

56—POWER TRANSMISSION: Bulletin A-660 issued by *Dodge Mfg. Corp.* explains and illustrates rolling bearings, sheaves and belts, shaft hangers and collars, couplings, and conveyor pulleys, to mention a few.

57—HEAT TREATING: Universal heat treating unit, fully automatic, sealed, and atmosphere controlled is described in brochure available from *Ipsen Industries, Inc.* Cross-sectional diagram and specifications are included.

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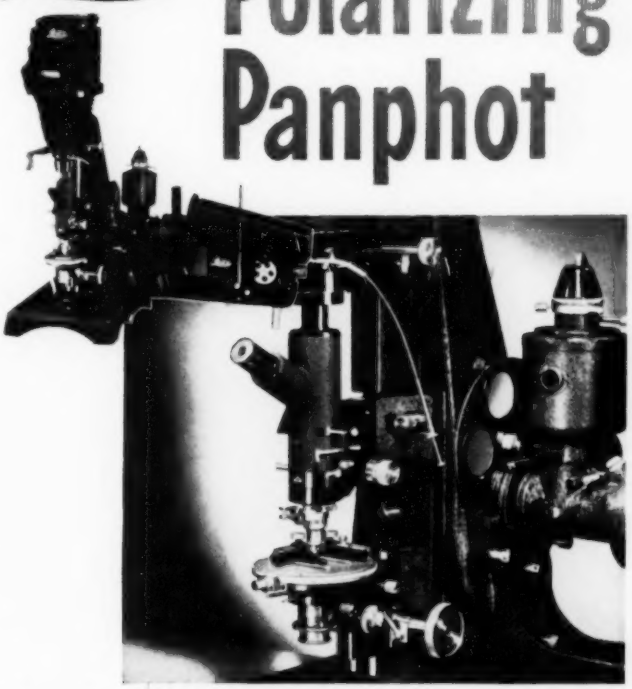

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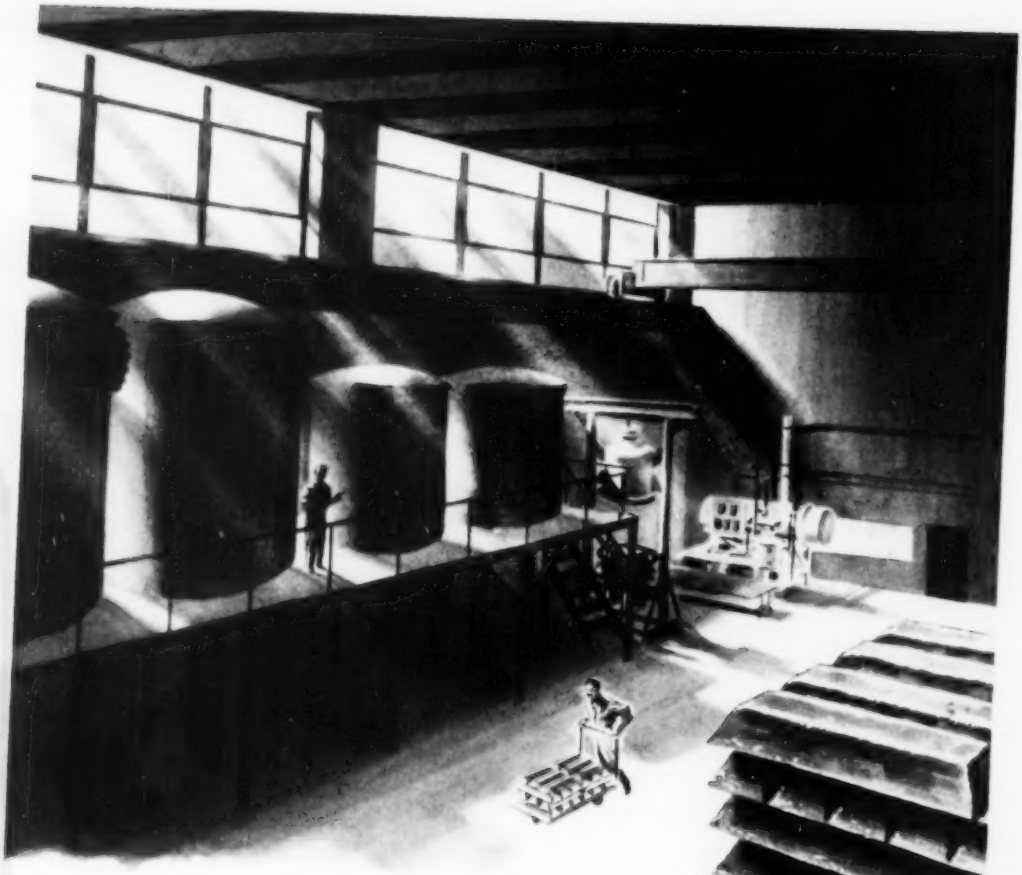

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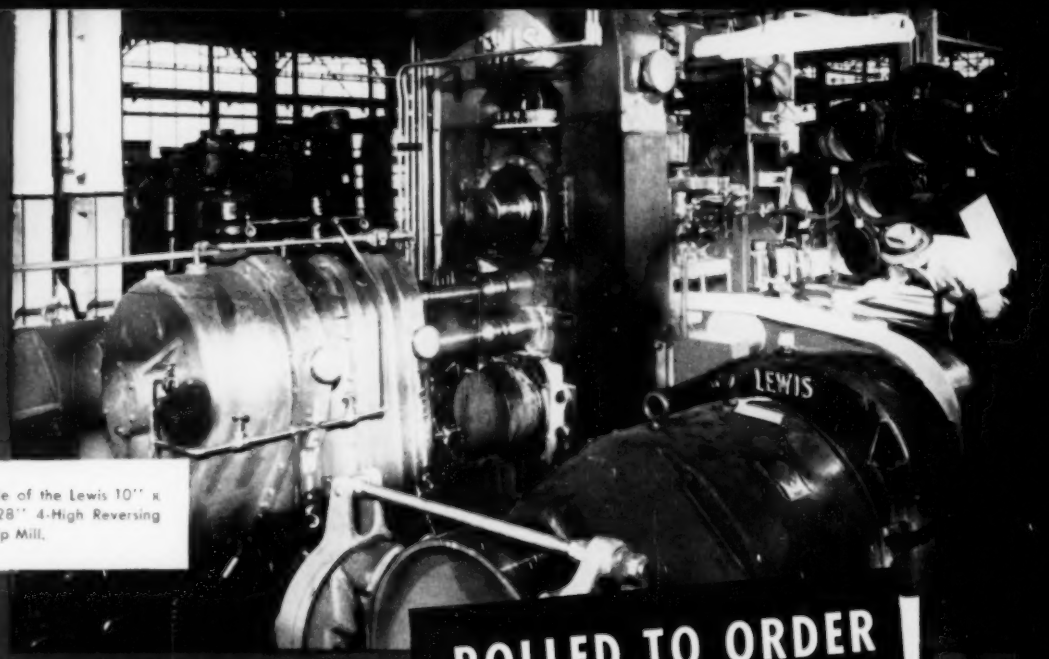

Entry side of the Lewis 10" x 20" x 28" 4-High Reversing Cold Strip Mill.

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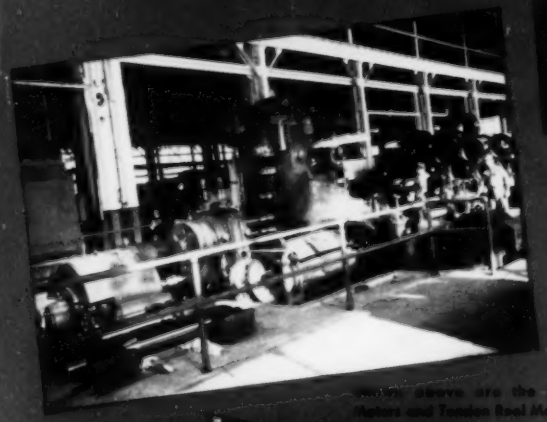

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Constant tension over full coil build-up and Mill speed range is effected through this Control Panel.

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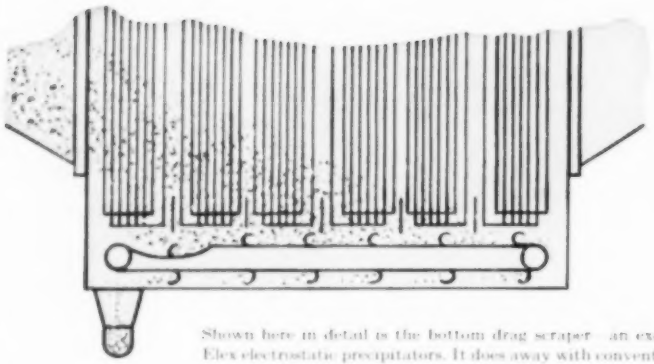

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Shown here in detail is the bottom drag scraper—an exclusive feature of Koppers-Elex electrostatic precipitators. It does away with conventional hoppers and simplifies operation by removing dust continuously.

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To get maximum recovery and effective nuisance abatement, specify Koppers-Elex electrostatic precipitators. For the metallurgical field Koppers has two distinct designs: the dry dust precipitator for hearth, flash, fluidized roasters, etc. . . . and the acid mist precipitator for secondary cleaning.

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— Coming Events —

June 2-4. Chemical Institute of Canada, annual conference, Montreal.

June 4-14. Mechanical Handling Exhibition and Convention, Olympia, England.

June 6. AIME, Columbia Section, dinner meeting, Spokane Hotel, Spokane, Wash.

June 16-20. ASME, semiannual meeting, Sheraton-Gibson Hotel, Cincinnati.

June 16-20. Industrial Finishing Exposition, International Amphitheatre, Chicago.

June 20-21. AIME, Central Appalachian Section, West Virginia Coal Mining Institute, joint meeting, Prichard Hotel, Huntington, W. Va.

June 22-26. International Powder Metallurgy Seminar, Reutte, Tyrol, Austria.

June 23-27. ASTM, 50th anniversary meeting, Hotel Statler, New York.

June 24-28. Industrial Physics Conference, Royal Technical College, Glasgow, Scotland.

July 5-Sept. 30. Centennial of Engineering, Chicago.

Aug. 25-29. Oak Ridge Summer Symposium, Oak Ridge, Tenn.

Sept. 3-6. AIME, fall meeting, Palmer House, Chicago.

Sept. 3-6. AIME, Industrial Minerals Div., annual fall regional meeting, Chicago.

Sept. 3-13. AIME, Chicago Section, Centennial of Engineering, Chicago.

Sept. 10-12. Porcelain Enamel Institute, annual shop practice forum, University of Illinois, Palmer House, Chicago.

Sept. 11-13. American Institute of Chemical Engineers, Palmer House, Chicago.

Sept. 22-25. American Mining Congress, Metal and Nonmetallic Mining Convention and Exposition, public auditorium, Denver.

Sept. 24-25. Institution of Mining and Metallurgy, symposium on mineral dressing, Royal School of Mines, London.

Oct. 3. AIME, Southern Ohio Section, National Open Hearth Steel Committee, Deshler-Wallick Hotel, Columbus, Ohio.

Oct. 10. AIME, Eastern Section, National Open Hearth Steel Committee, Warwick Hotel, Philadelphia.

Oct. 10. AIME, Southwestern Section, National Open Hearth Steel Committee, Peoria, Ill.

Oct. 20-22. AIME, Institute of Metals Div., fall meeting with National Metal Congress, Hotel Adelphia, Philadelphia.

Oct. 30-31. AIME, Fuels Conference: Coal Div., AIME; Fuels Div., ASME, Bellevue-Stratford, Philadelphia.

Nov. 7. AIME, Pittsburgh Section, National Open Hearth Steel Committee, Hotel William Penn, Pittsburgh.

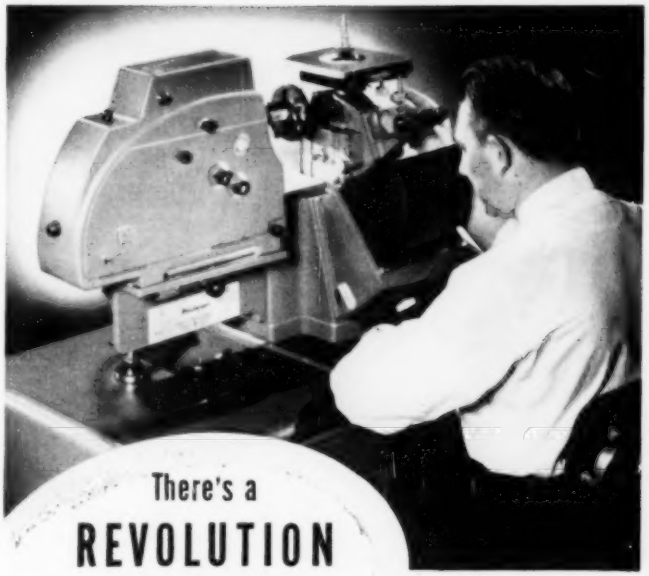
Nov. 18. AIME, Buffalo Section, National Open Hearth Steel Committee, Hotel Statler, Buffalo.

Dec. 1-6. AIME, Electric Furnace Steel Conference, Hotel William Penn, Pittsburgh.

Dec. 8. AIME, Arizona Section, all day meeting, Tucson.

Feb. 16-19, 1953. AIME, annual meeting, Statler Hotel, Los Angeles.

Apr. 20-22. AIME, National Open Hearth and Blast Furnace, Coke Oven and Raw Materials Conference, Hotel Statler, Buffalo.



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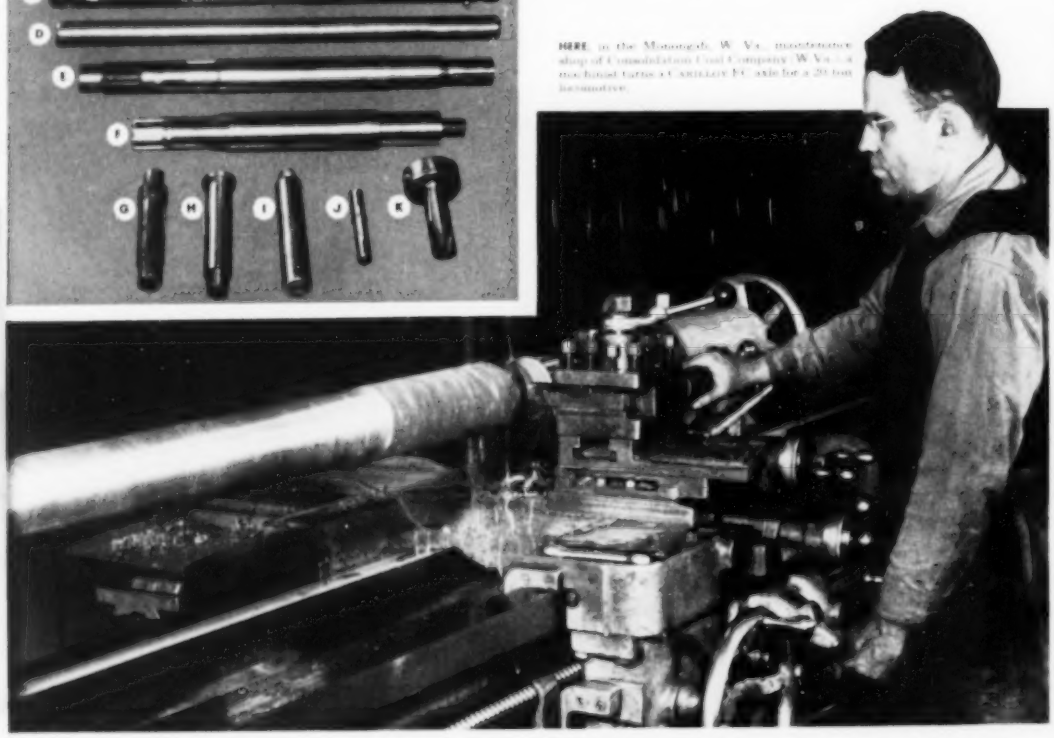
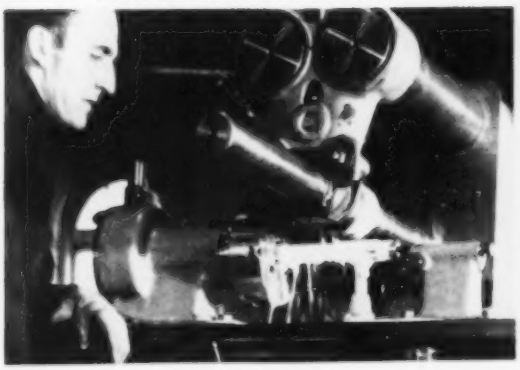
CARILLOY FC is used in machining this great variety of parts. Notice the diversity of applications handled by one grade of alloy steel. (A) is an armature shaft. The following parts are for loading machine; (B) loading clutch shaft, (C) conveyor take-up shaft, (D) conveyor hinge pin, (E) transmission clutch shaft, (F) caterpillar drive axle, (G) caterpillar idler axle, (H) sprocket shaft, (I) caterpillar take-up shaft, (J) hydraulic pump shaft, (K) caterpillar take-up screw.

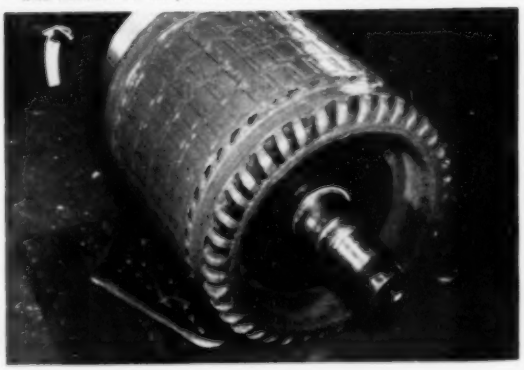
HERE, in the Monongah, W. Va., maintenance shop of Consolidation Coal Company (W.Va.), a machinist turns a CARILLOY FC axle for a 20-ton locomotive.



CARILLOY speeds up milling of a loading machine shaft.



THE SHAFT for this 1900-pound locomotive armature was machined from CARILLOY FC and put into service without heat treatment.



made without heat treating

with pre-hardened free-cutting

CARILLOY FC STEEL

EVERY year, over 5 million tons of coal pour out of the 6 Fairmont, West Virginia, region mines of Consolidation Coal Company (W. Va.). All repair and rebuilding of the hard-working equipment used in these mines are done at the company's Monongah, W. Va., maintenance shop.

Here, highly skilled machinists make everything from mine locomotive axles to armature shafts. Each new part they make must be as strong as the original, without benefit of heat treatment after machining.

So here is just the place for a very special kind of alloy steel.

Consolidation Coal Company (W. Va.) has found that they can produce practically all of the heavily stressed parts they use in their own shops—without heat treating—with a special grade of alloy steel, such as U·S·S CARILLOY FC steel. This pre-hardened, free-cutting alloy steel is received from the mill already quenched and tempered to the hardness required. No further heat treatment is necessary. Finished parts have a minimum tensile strength of 140,000 psi. and good surface finish.

U·S·S CARILLOY FC offers you important advantages if you have to make heavy-duty shafting or other parts that require 125,000 to 175,000 psi. steel. This deep-hardening manganese, chromium, molybdenum alloy steel is available quenched and tempered from 255 to 375 Brinell. You don't have the expense of heat treating after machining and you eliminate rejects caused by distortion and scaling.

For *production* work, remember that CARILLOY FC, easy to machine, is easy on your tools—some users report that tools last 300% longer, and more pieces can be produced per hour.

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UNITED STATES STEEL

Books for Engineers

Thermodynamics of Alloys, by John Lumsden. *The Institute of Metals, London.* $5.00, 204 pp.—Dealing with the application of thermodynamics to the quantitative study of phase equilibria in metals and alloys, the emphasis is on the correlation of equilibrium properties. Theory is developed from first principles, with mathematics kept as simple as possible. Selected examples are used to illustrate how a comprehensive free-energy equation, derived from suitable experimental measurements can be used reliably to calculate all the equilibrium properties of a system.

Report on the Elevated Temperature Properties of Stainless Steels, prepared by Ward F. Simmons and Howard C. Cross, under the auspices ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals. *American Society for Testing Materials.* $4.00, 115 pp.—The report is essentially a graphical summary of elevated-temperature data for commercially produced stainless steels. It contains summary curves for tensile strength, 0.2 per cent offset yield strength, elongation, reduction of area, stresses for rupture in various time spans and stress for various creep rates. The appendix contains the primary data from which the curves were drawn, chemical composition, processing data and other pertinent information about the steels.

Foundry Practice, by William H. Salmon and Eric N. Simons. *Pitman Publishing Corp.* $6.50, 384 pp.—The book is a practical, comprehensive guide covering the field from blueprint to quality control, including patterns, molding and core-making, pouring, molten metal, the cast metal and heat treatment. The material is so arranged that it is suitable for adoption as a text book. It includes a question and answer section.

Practical Radiography for Industry, by H. R. Clauser. *Reinhold Publishing Corp.* $7.50, 301 pp.—The book is directed at those who actually perform radiographic inspections and persons who read or interpret radiographs and evaluate their findings. Equipment and fundamental principles are treated in concise, fairly nontechnical language, understandable to those with little or no training in physics or mathematics. The book contains nearly 200 illustrations including sample radiographs showing defects in metal parts. Information on procedures, exposure factors, techniques, quality, films and film processing is presented.

Extrusion of Plastics, Rubber and Metals, by Herbert R. Simonds, Archie J. Weith and William Schack. *Reinhold Publishing Corp.* $10.00, 434 pp.—One fourth of the book covers the extrusion of metals, rubber, and miscellaneous materials. The major portion of the volume is devoted to extrusion in the plastics industry. It includes a glossary and a brief buyer's guide.

Chill-Cast Tin Bronzes, by D. Hanson and W. T. Pell-Walpole. *Longmans, Green & Co.* $14.00, 368 pp.—The authors' aim is to provide a comprehensive view of bronze metallurgy with special reference to ingots and castings made in metal molds. A short historical survey and general treatment precedes two chapters on the melting process and three on the casting process. Theoretical and practical details are covered. Other chapters deal with segregation effect of minor constituents and solid impurities, heat treatment of cast bronze, and metallurgical aspects of wrought bronzes. The properties of cast bronzes are listed and an extensive bibliography included.

Diffusion in Solids, Liquids, Gases, by W. Jost. *Academic Free Press, Inc.* $12.00, 558 pp.—The factors influencing diffusion in all types of bulk phases and across a number of interfaces between bulk phases are dealt with by the writers. Emphasis is on diffusion in solids. The book deals with fundamental laws of diffusion, disorder in crystals, solubility in solids, permeation and diffusion of gases in solids, surface reaction of metals, diffusion in gases and liquids, and thermal diffusion.

Chemical and Electro Plated Finishes, by H. Silman. *Chapman & Hall, Ltd., London.* 50s, 473 pp.—The author has gathered a reasonably comprehensive account of modern industrial metal finishing processes, with the chemical and physical principles involved. The book will be of special interest to engineers, designers and those concerned with the scientific and technical side of metal finishing. Included are all finishes applied to metals for both protective and decorative reasons from aqueous solutions with or without electric current.

Bibliographic Survey of Corrosion, by the Engineering and Publishing Staffs, *National Assn. of Corrosion Engineers.* $9.00, 288 pp.—A comprehensive bibliography compiled from major abstracting services as well as from a range of specialized indexing sources. The items are arranged according to a classification system devised by the NACE, based on careful subdivision of eight major sections. General testing, corrosion types and influencing factors, corrosive environments, preventative measures, construction materials, equipment, and industries. There is also an authors' index, a subject guide, and notes on means for securing indexed articles.

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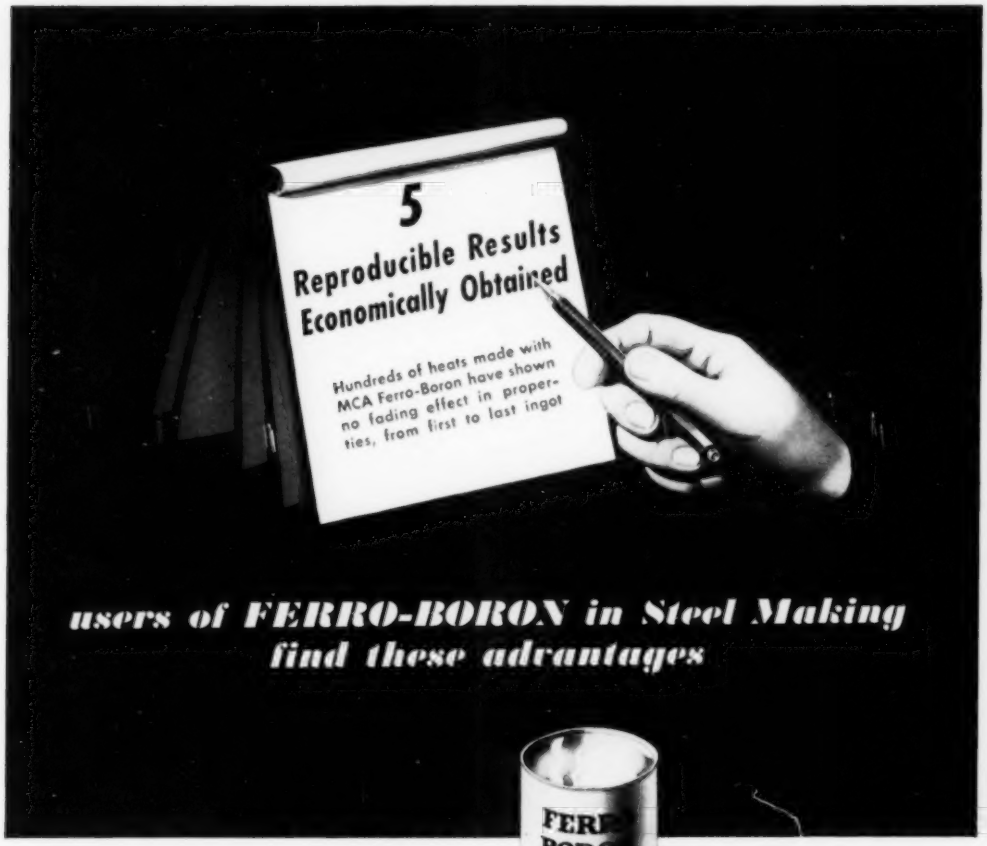
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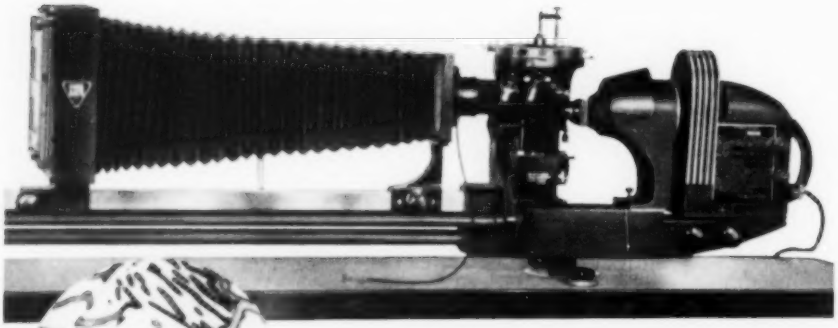

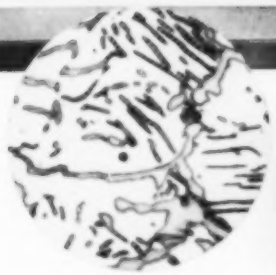

MICRO and MACRO

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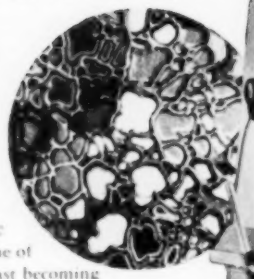

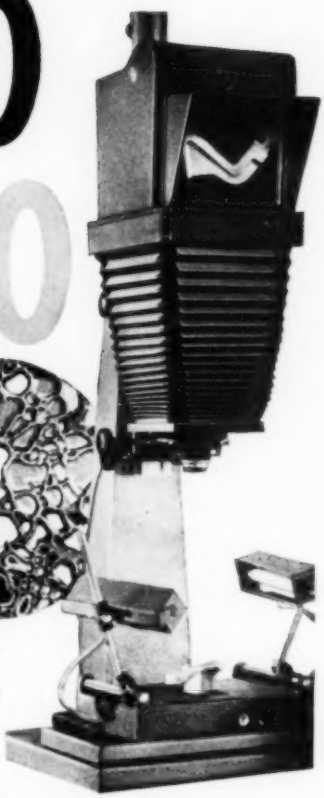

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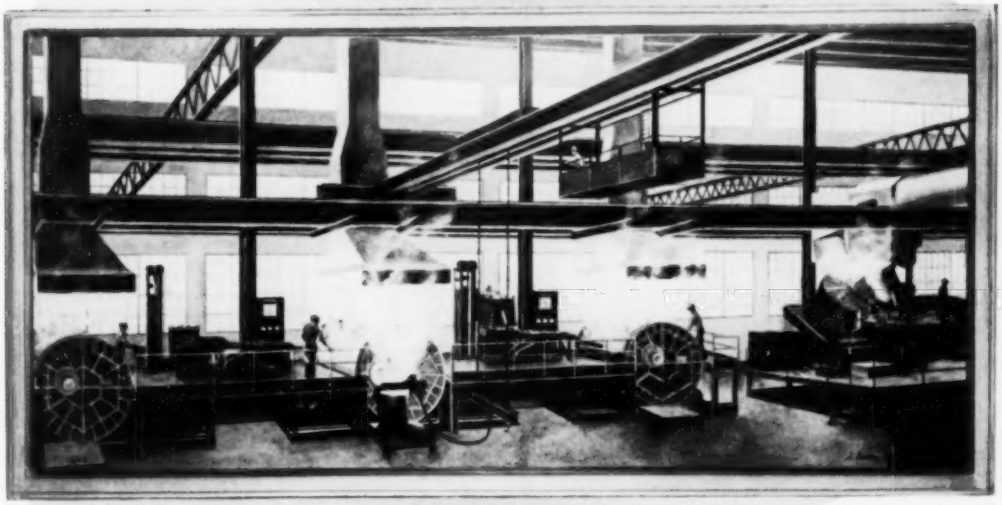

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Every half hour each of these furnaces in turn discharges its 5,000-pound charge into a ladle which is transferred by crane to a holding furnace (shown at right) of 9,000 pounds' capacity. This furnace is mounted over the continuous casting machine located on the floors below, and thus is able to provide a permanent flow of molten brass to the mold. The intermixing of the charges from the three furnaces gives additional uniformity of composition.

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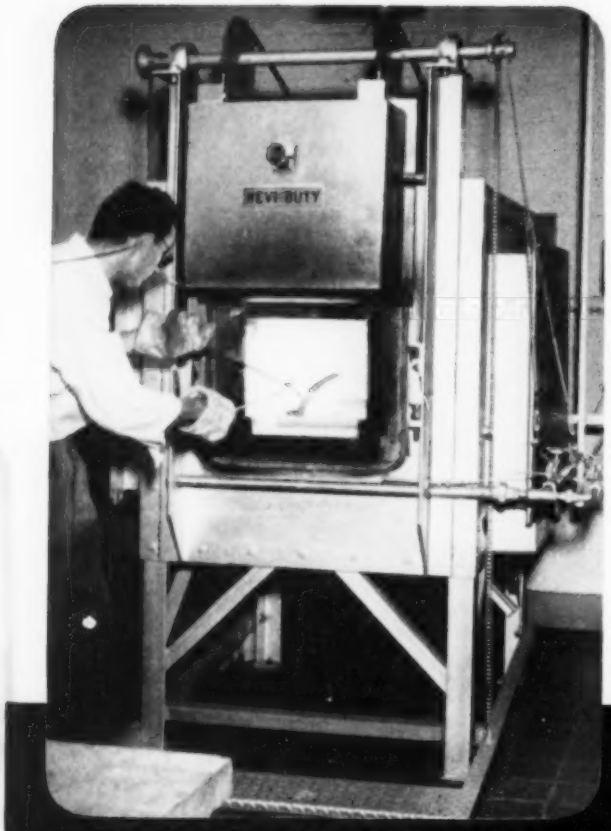

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Industrial Notes . .

• The Aluminum Co. of Canada and the United States are continuing contract negotiations started some time ago. Alcan's original stipulation was for a guaranteed market of 900,000 tons, but recently has modified its terms to 400,000 tons. The contract would be in line with top ranking DPA official Samuel Anderson's aluminum expansion program calling for increased domestic facilities and a boost in Canadian imports.

• **Rimmed steel deoxidation is being accomplished with the aid of rasorite, a sodium borate concentrate commonly used in kitchens under the name of borax. It has been increasingly replacing or supplementing aluminum in recent months.**

• Mercantile Metal & Ore Corp., formerly of 60 Wall St., New York, has moved to larger quarters at 595 Madison Ave., New York. Mercantile Metal, with associated firms in Europe, South America, and the Far East, trades internationally in minerals, metals, and ores.

• Britain imported 172,000 long tons of steel, pig iron, and scrap from the United States during the first quarter of 1952. In a report to the House of Commons, Supply Minister Duncan Sandys said American purchasers are "holding off" buying in other countries in an effort to enlarge the supply available to Britain. He also stated, in reply to questioning by a left wing Labor party member of the House, that Russia could serve as an alternate supplier.

• *Armco Steel Corp., Middletown, Ohio, has suspended plans for $45 to $50 million worth of expansion. The company gave high taxes and the uncertain labor conditions as the reason. However, construction already in progress will be continued, the company spokesman said.*

• Belgium steel production jumped more than 128,000 tons for the first quarter of 1952, compared with the same period one year ago. Five million tons were produced for the first time in the country's history during 1951. Trade sources feel that Belgium can produce 5.4 million tons per year. The industry now has 51 blast furnaces, 51 Thomas converters, 28 Martin ovens, and 37 electric ovens. More than 50,000 men are employed.

• **The first production of .50 calibre cartridges has been achieved from a billet of specially processed sintered iron. Although the ex-**

periment did not result in a finished product, experts said it showed the possibility of making such cartridge cases.

• Curtis-Wright is planning a $5 million jet parts expansion program at Buffalo, with the possibility that the investment may be raised to $10 million. The company said the additional outlay would depend on negotiations with the Air Corps for manufacture of propeller blades by extrusion.

• *The first steel plant to be constructed in Egypt will be financed by a group of German firms. The plant will have a capacity of 115,000 tons annually. The group has been awarded an exclusive Government contract worth about $51.6 million.*

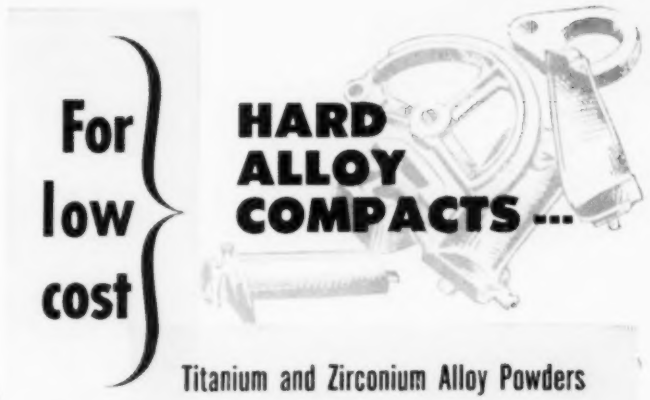
• Birsboro Steel Foundry & Machine Co., is forming a new subsidiary, Birdsboro Armorcast, Inc., to make tank hull and turret armor castings.

• **An Army Signal Corps report prepared by the Georgia Institute of Technology describes a new fatty acid absorption technique for surface area measurement of fine particles. Of particular use in metallurgy, the process greatly reduces measurement time, and can be widely used to control chemical processes.**

• The Duquesne Works of U. S. Steel is installing nine new scrubbers to wash blast furnace gas. The installations are expected to greatly reduce dust content in furnace gases. A water spray inside of the washers is diffused through stainless steel screens. The washed gas is recovered for use in the plant's boilers. Recovered iron ore dust is sent to sintering plants. Installation is expected to take about a year.

• **Canadian Siempelkamp, Ltd., plans to manufacture industrial presses heavier than any now made on the North American continent, in a plant to be constructed at Hamilton, Ont. The new company expects orders from South America, in addition to those from the United States and Canada.**

• The Air Force Experimental plant at Adrian, Mich., has installed the world's largest neutral salt bath. The bath operates at a temperature of 2350° and is being used to heat steel billets for press forging. The 128,000 lb of molten salt are kept neutral by Park Chemical Co.'s Neutro-Gas Process. The bath is 20 ft long, 10 ft deep, and 4 ft wide and is a 5000 kw capacity electric furnace. The gas purifying tanks are 12 ft long. Bohn Aluminum operates the plant for the AF.



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THE DRIFT OF THINGS

by Edward H. Robie

EIGHT years ago in this department we had a column on the daiquiri cocktail, which, as we pointed out at that time, was invented by mining engineers. Since then we have said little or nothing about alcoholic drinks, but having set the precedent perhaps we may now pass along a little information about martinis.

We have observed that as an aperitif the martini has become increasingly popular with men, so much so that we always feel a bit peculiar if we order something else. And the call is not for just a martini either; it must be a *dry* martini, and some of our friends are extremely particular on this point. The matter has achieved such importance that *The New York Times* devoted half a page to it a few Sundays back. Several recipes are given which may be useful to some of our members when entertaining visitors from this part of the country.

The standard martini is said to be half gin, half Italian vermouth, two dashes orange bitters, and a pickled onion and twist of lemon peel. This progresses to the *very dry* martini, which calls for five parts gin and one part vermouth with an onion, olive, or lemon peel. Several alternatives are offered for the manufacture of the *very, very dry* martini. One is to pour a little vermouth into the mixing glass and pour it out again before mixing gin and ice. A second way is to put the ice in a sieve, hold it over the sink, and pour a little vermouth over it; then put the ice in the mixing glass. A third is to let the draft from an electric fan blow across an open vermouth bottle toward the mixing glass. Then the ultimate is the *awfully dry* martini, obtained by setting the vermouth bottle next to the mixing glass and turning the bottle slowly so that the label is exposed to the gin for perhaps a second.

The Times notes that Mr. Martini himself personally prefers vermouth and soda.

Be Sure and Vote

We understand that a certain number of AIME members read this department to the exclusion of all else in the journals. We even have fairly definite proof that this statement applies to 0.05 pct of the members. For the benefit of this possibly small but undoubtedly select group, we want to call attention to the statement about the dues referendum that appears on page 670 of this issue.

Now of course this Institute, which has been running along through good times and bad for 81 years, will not go out of existence if the Member dues go back to $15 and the Junior dues to $10. But it seems fairly certain that the Directors will, in fact cannot, take a chance on a deficit next year, so in one way or another you would get less than you get now. So it boils down to what you want. A $20 or a $15 Institute, or even a $10 Institute would be possible if AIME members were willing to forego most of the things that have made the AIME what it is. On the other hand, it is entirely possible that if we did as the ASME recently did, and raised our dues to $25, or as the Engineers Society of Western Pennsylvania did and raised them to $30, we could give our members so much more that they would think the increase a good investment. We do not think it would ever be advisable, though, to raise our initiation fees and dues to the levels of some of the labor unions!

Ian Campbell wonders if we have called attention to the fact that the present $20 dues are really no more than the old $15 dues because they can be deducted from income for tax purposes and most of our members are in the 25 to 30 pct tax category. We explained this in some detail in the Drift for March 1949. But as some of our younger members have pointed out, the argument falls down when the taxpayer takes a flat 10 pct deduction and does not itemize his business expenses and contributions.

Your ballot should reach you before the end of June (it goes only to members in North America) and should be returned right away. Like the politicians, we urge every one to vote. Particularly if they vote right. Three fifths of the members voted in the last referendum. Let's do better this time.

Birthday Greetings

AIME's oldest living member was born on July 3, 1849, so next month will be 103 years old. He is Dr. Edward A. Uehling, of 2360 South 81st St., West Allis 14, Wis. We have a letter from him dated March 21 which says, "The years and weather keep me confined to my room. I extend hearty greetings to all the members of the Institute." We think it would be nice if some of our members responded to this greeting. Dr. Uehling joined the Institute 66 years ago, nine years after he graduated from Stevens Institute. His work was in iron and steel, and 65 years ago he was superintendent of blast furnaces for the Bethlehem Iron Co., at Bethlehem, Pa. He then became furnace manager for the Sloss Iron & Steel Co. at Birmingham, Ala. Later he developed and introduced a pig iron casting machine and a pyrometer. He was one of eleven children and his mother lived to be 99.

Engineers' Beatrice Fairfax

Among our extracurricular duties this year is acting as secretary for the Engineers Council for Professional Development. In this capacity, our attention was recently called to a short piece in the March issue of *Family Circle*, a 5c magazine sold at Supermarkets. It was in a department headed "Teen Scene" and it told of the dire need for engineers, and urged those who are good at math and science to give serious thought to preparing for the engineering profession. It wound up by saying that those interested should write to ECPD. Close to 100 inquiries are lying on our desk as we write this.

Most of these require thoughtful and intelligent answers, but some are merely amusing. For instance, a young lady in Gallion, Ala. writes:

"Dear Engineers:

"I'm a teen age Girl. I'm fast losing my friends and wonder what can I do to make him understand. Please Send me Some empermashion. I also quiet my best Friend, the one I really love. What can I do to get him back gain? And I want him for my husband."

And this from McCorkle, W. Va.:

"Dear Engineers

"I am 13 years old and I am in Love with a boy and he is 18 years old and he youst to Like me a lot but he go With another girls But seems he Likes me the Best of all the girls, I Just Don't no how to take him. Would you go with him or not."

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National Lead Co. and Fomento Minerales de Cuba have bought the 50 pct interest owned by N. V. Billiton in Nickel Processing Co., operators of the United States Government owned plant at Nicaro, Cuba. National Lead gains the controlling interest in view of its original 30 pct participation in the project. Fomento, before Billiton was bought out, owned 20 pct of Nickel Processing. Further expansion of production is contemplated with a contract for pilot plant construction awarded. Part of the deal with Billiton included rights to certain processes that are expected to raise nickel output and to give cobalt as a byproduct.

An increase in German steel production during the early part of the spring lifted plant output to an annual rate of 15.8 million (metric) tons. Previous speculation had placed Germany's expected production at 15 million tons. Partially responsible for the gains is the increase in German coal production. Pig iron production hit 1.058 million tons, up 70,000 tons from February.

The United Nations Economic Commission for Europe warns that steel producers on the continent must expand and modernize if competition from the United States and Japan is to be met successfully. Predicting that Japanese and American competition will grow stiffer within the next few years, the Commission warned that European producers could not depend upon arms contracts to maintain them under long term conditions.

General Electric will use aluminum bases for 50 pct of its electric light bulb production in 1952.

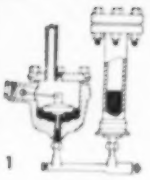
American taxpayers are being taken off the hook to the extent of $2 million with the adoption of certain powder metal parts by Army Ordnance. Further utilization of powder metal may increase the savings to $10 million. If and when powdered iron rotating bands come into use, an additional $8 million may be saved.

Estimate of surplus zinc supply for American needs is placed at 139,000 tons for 1952 by C. R. Ince, vice-president of the St. Joseph Lead Co. He figured industrial needs at 889,000 tons.

Steel is being produced by a new process almost totally different from conventional bessemer and open hearth procedures, at a pilot plant at Oakville, Ont. The method known as the Cavanaugh process, utilizes a kiln. Test slabs are being shipped to Republic Steel, where they are being rolled into sheets. Manufacture of controlled density steel is expected to take root in areas where there is little capital for regular steel plant operation or the know-how is lacking.

Government and industrial officials are off to Europe to buy large steel castings needed for the heavy press program sponsored by the Air Force. The team will place orders for 69 separate castings ranging from 50 to 150 tons. The few American installations capable of producing the castings are already overburdened by defense orders. Eight units called for are forging presses with applied pressure capacity ranging from 25,000 to 50,000 tons. Another nine are extrusion presses ranging from 8000 to 20,000 tons.

Losses caused by conventional methods for feeding cast iron borings into the cupola in making pig iron, may be drastically curtailed by a new British method whereby the metal scrap is fed through a hole in the side directly into the heat zone, instead of poured through the top. The new unit is said to be capable of saving 1 million tons of cast iron annually.



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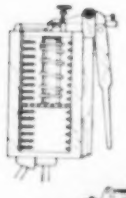

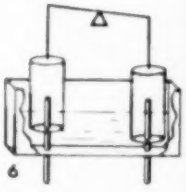

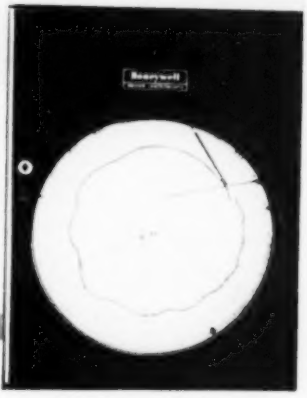

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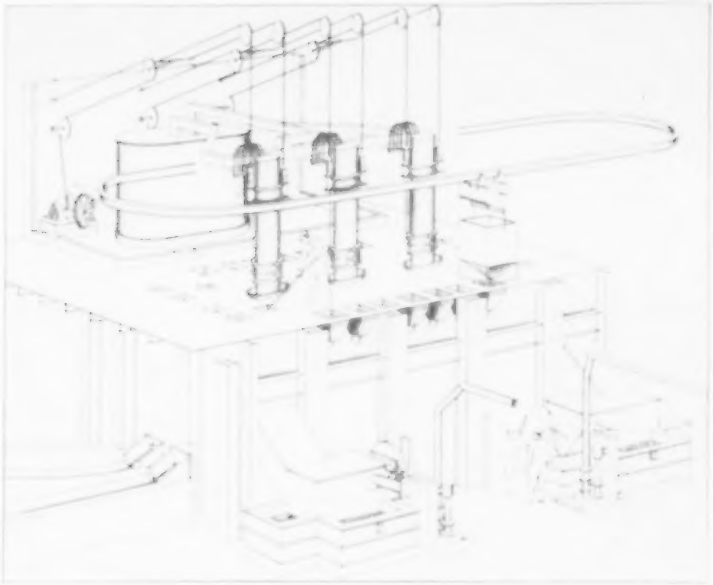

Fig. 1—The heart of the Sterling Process is the use of an electric arc to carry on reduction of zinc ores. This is a schematic diagram of the electric furnace used.

Electric Furnace Smelting of Zinc Ores

The Sterling Process

by E. C. Handwerk, G. T. Mahler, and L. D. Fetterolf

The Sterling Process is a new method for smelting zinc ores using heat generated by an electric arc to carry on the reduction. Covering patents have been allowed to the New Jersey Zinc Co. By the process, zinc metal, together with its accompanying values, and pig iron are produced. All of these elements except for a controlled amount of iron are almost completely eliminated from the slag. The process derives its name from the fact that initially it was directed to the smelting of the crude ore from the New Jersey Zinc Co.'s Sterling Hill mine, which ore, after calcining, assays 18 to 20 pct zinc and an equal amount of iron. It has since been used on high grade zinc concentrates from which substantial recovery of secondary values can be realized. The lead, cadmium, and some of the silver, are collected in the zinc metal, while copper, gold, and the remaining silver are concentrated in the pig iron. This article is the first to describe the theory and operation of the patented Sterling Process.

THE idea of smelting zinc with the use of a heat supply derived from electric energy arose as soon as electric furnaces were developed. The early art, as set forth in a large number of patents, suggests the use of every known type of electric furnace for smelting zinc. The development followed two lines.

In one, the charge of the zinc furnace was used as a resistor and smelting was effected by the heat generated in passing an electric current through the charge. The charge was not melted and no slag was produced. This method of using energy to smelt zinc ore was the first to be successfully practiced on a large scale. St. Joseph Lead Co.[1] developed a process for smelting by electric resistance a preheated, sintered zinc ore charge in large vertical retorts, discharging a solid unfused residue.

The second line of development was the use of an electric furnace in which higher temperatures were developed by arc and slag resistance, the charge being completely melted down and the residue discharged as slag. In attempting to operate early electric furnaces in this manner, satisfactory condensation of zinc was a major problem. The difficulty was that the zinc vapor did not condense predominantly to liquid zinc but instead to zinc dust or blue powder. Many investigators became resigned to accepting the formation of blue powder and operating the electric furnace so as to form zinc dust only, which was then melted down to liquid zinc in a separate operation. This idea was ultimately carried out in the most persistent attempt to produce zinc in electric furnaces, namely, the Trollhättan electrothermic zinc process. This work—which was carried on at

E. C. HANDWERK, G. T. MAHLER, and L. D. FETTEROLF are associated with the New Jersey Zinc Co. [of Pa.], Palmerton, Pa.

Trollhattan, Sweden, and Sarpsborg, Norway, more or less continuously for some 40 years—has been described in the literature.

The situation with respect to smelting zinc ores in open arc electric furnaces before the Sterling Process entered the field may be summarized briefly.

A number of ideas had been developed that now have proved to be sound, but heretofore it was not known how to apply these ideas successfully in operating a furnace. Moreover, no idea had been put forward for controlling heat distribution in the furnace so that substantial reduction of zinc and iron is accomplished prior to slagging and so that volatilization of constituents interfering with condensation is avoided. The absence of an effective method for condensing large volumes of hot dilute zinc vapor without formation of excessive amounts of blue powder greatly interfered with previous experimental work and rendered it difficult to evaluate the suggested ideas. However, it should be evident from the following discussions that adequate condensing was only the beginning of the story.

Theoretical Considerations

One of the basic elements of the Sterling Process is the electric furnace reduction of a charge consisting of oxidic zinc ore or concentrates and carbonaceous material, so managed that most of the zinc content of the charge is reduced before the gangue melts to a slag. The actual zinc reduction thus occurs predominantly before fusion of the gangue, so that the difficulty of eliminating zinc from molten slag is largely avoided. Another fundamental feature of the Sterling Process is the prevention of direct contact of the electric arc with solid charge—an important feature, since an arc impinging upon the charge produces quantities of dust that interfere with subsequent condensation of the zinc vapor to molten zinc.

Broadly speaking, there are two methods of operating in accordance with these principles.

The first is to operate with an arc submerged in the molten slag. When operating in this manner, heat adequate for smelting is transferred to the solid charge through the slag, so that zinc reduction occurs principally at the interface between the molten slag and the solid charge. With proper control of slag fluidity, readily condensable zinc vapor can be obtained. However, the method involves supplying heat in relatively large amounts to a mobile slag, and consequently there is a risk of damaging the sidewalls of the furnace.

According to the second method, the furnace is heated by open arcs playing from the electrodes to the surface of the slag and not impinging upon the solid charge. Heat is supplied to the charge largely by direct and indirect radiation from the arc, so that zinc is eliminated from the solid charge largely at the surface of the charge, before it melts down to slag.

In actual practice, heat is applied by a combination of both methods, but it is preferable to provide the heat for zinc reduction predominantly by open arc radiation.

The theoretical metallurgy of zinc has been well described in the literature[1] but a brief review of the zinc smelting reactions may be helpful in describing the Sterling Process and the factors that govern its successful operation.

When a mixture of ZnO and C is heated, ZnO is reduced, forming Zn vapor and CO. CO, the active reducing agent, is regenerated from CO_2 by reaction with C. These reactions are reversible. Any CO remaining in the reaction mixture will reoxidize Zn vapor during cooling. Fortunately, CO does not revert to CO_2 and C at a significant rate except in the presence of catalysts. The reduction reaction takes place at a practical rate in the neighborhood of 1050°C. This is above the boiling point of zinc and zinc therefore is evolved as a vapor.

In the presence of C, the CO content decreases with increasing temperature, and it is therefore desirable to have the reaction mixture leave the reacting materials at as high a temperature as possible. In the ordinary zinc reduction processes, temperatures of 1050°C or somewhat above are usual and, since this is below the slagging temperature of the residue, a dry residue is left. An essential difference in the Sterling Process is that the temperatures involved are above the melting point of the residue and the residue is removed from the process as a molten slag.

Factors to Be Controlled

In the Sterling Process, it is most essential that the correct amount of reduction fuel be charged to the furnace. The theoretically required amount of carbon is added to reduce the zinc, lead, cadmium, and part of the iron in the charge. It has been found advisable to leave 1.5 to 6 pct of the iron unreduced as a component of the slag for two reasons. At the high temperature of the arc, it is possible to reduce and/or volatilize lime, silica, and magnesia from the slag into the furnace atmosphere where they form nuclei on which zinc vapor can condense to form blue powder. If some iron oxide is present in the slag, it is reduced preferentially with respect to the other slag constituents in the proximity of the arc. On the other hand, if the iron oxide content of the slag is allowed to go much over 6 pct, it will decarburize the molten pig iron in the furnace and produce an iron which cannot be tapped satisfactorily at the prevailing furnace temperature. Thus it can be seen that, by controlling the amount of carbon in the charge, the iron oxide content of the slag can be held in the range necessary for good operation. In addition, it is necessary to avoid excess coal which would accumulate and interfere with furnace operation.

The composition of the slag in the Sterling furnace must be adjusted carefully so that the desired fluidity is obtained at operating temperatures. By the addition of lime or silica, depending upon the ore constituents, the CaO:SiO$_2$ ratio is maintained in the range of 0.8:1 to 1.4:1. This allows sufficient fluidity at 1300° to 1350°C for the reduced iron globules to settle through the slag and form a pool on the bottom, and permits easy tapping of the slag. In addition, slag fluidity and mobility are necessary to aid in distributing heat from the arcs to the charge and the molten iron.

It is essential that the gases passing from the furnace to the condenser be as low in CO content as possible. Furthermore, any unnecessary dilution should be avoided. Preheating the charge is beneficial in two ways. First, by partially reducing the higher oxides of iron, dilution is decreased. Second, contact of the zinc vapor–CO mixture with cold charge as the gases leave the furnace is avoided. Such contact favors reversion of CO to CO_2. These advantages are in addition to the saving in power.

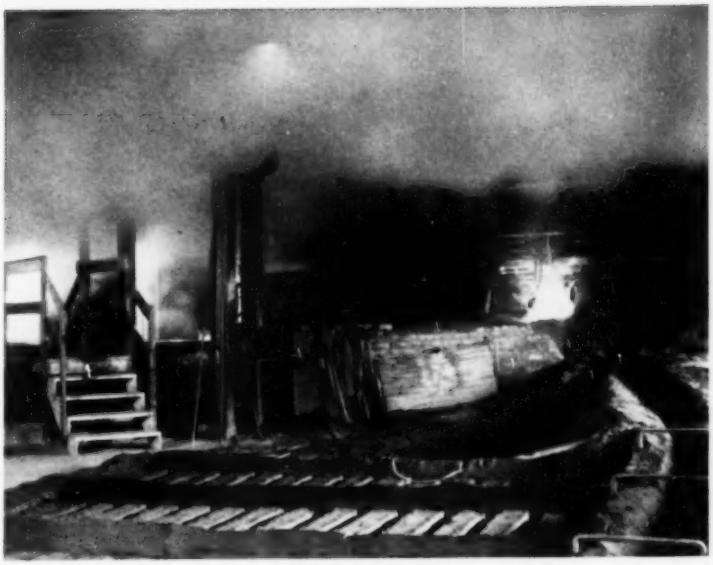

Fig 2—Slag has been tapped from the furnace and is flowing through the runner on the left where it is being granulated and cooled by water jets. The sand bed in foreground is ready for iron to be tapped out of the lower tap hole.

A major problem which has been solved in developing the Sterling Process is the practical utilization of the intense heat of the electric arc. While the arc temperature is 3000° to 4000° C, it is desirable to maintain the bulk of the furnace contents below 1450° C to avoid reducing the gangue constituents and overheating the refractories. Such low furnace temperatures are possible because the charge banks rapidly absorb the radiated heat for the endothermic reaction. The highly fluid, agitated slag bath also functions to dissipate some of the heat from the arc and distribute it to the charge floating on its surface where it is consumed in the reduction reaction. The high temperature zone is confined to the center of the furnace and refractory attack is minimized. Furthermore, by establishing steady conditions of charging rate and power input, the reaction is controlled so that the flow of furnace gases to the condensers proceeds at a constant rate conducive to good condenser efficiency.

Control of condensing temperature is the final requirement for efficient operation of the Sterling Process. In the new splash type condensers, the gases pass through a shower of molten zinc maintained at 500° C by water-cooled coils immersed in the liquid zinc bath. The condenser walls are insulated rather than cooled, so that essentially all of the heat liberated in cooling the gas and condensing the zinc vapor is absorbed by the dispersed molten zinc at 500° C. The intensive scrubbing by liquid metal is such as to effect maximum condensation of the condensable metal vapors as liquid metal rather than as solid blue powder.

The development which led to the understanding of the principles described and their practical embodiment in the Sterling Process, resulted from the desire of the New Jersey Zinc Co. to find a better method for recovering the metal values from Sterling Hill ore.

The first investigations were made on a reverberatory furnace in which the aim was to smelt run-of-mine Sterling Hill ore directly, to recover the zinc as an oxidized fume concentrate, the iron as pig iron, and the residue as molten slag. These results were encouraging and demonstrated the possibility of slag smelting a zinc ore on a large scale, with practically complete elimination and recovery of the zinc, together with the production of a fluid slag and pig iron.

The success of this work led to an investigation of slag smelting in an electric furnace, the newly developed, splash type condenser being used to collect the zinc in liquid form. It was during this experimental work with small electric furnaces that the principles described were established and successfully put into operation. After some experimentation with electric furnaces of progressively larger sizes, a rectangular furnace having internal dimensions of 16x32 ft was constructed at Palmerton, Pa. A schematic diagram of the furnace is given in Fig. 1. Fig. 2 shows slag flowing from the furnace, which can be seen in the background. The furnace is constructed of high grade firebrick with 24-in. thick sidewalls, a 3-course, inverted arch bottom, and a 15-in. thick, arched roof. At the crown of the arch, the inside height of the furnace is 10 ft. The bottom of the furnace is cooled by air, and the sidewalls are water-cooled. The furnace is heated with three 24-in. diam graphite electrodes in line along the arch, which are operated on 3-phase circuit. The furnace is provided with 6000-kw transformer capacity, although maximum sustained input to the transformer up to the present time has been 4500 kw. This has been due to lack of auxiliary facilities for handling the charge and the products, rather than to limitations of the furnace itself.

Description of the Process

As indicated in the flowsheet, Fig. 3, the oxidized zinc bearing materials, reducing fuel, and flux are carefully proportioned and thoroughly blended. The zinc materials, coal, and flux do not have to be dry for use in the process—as long as their moisture content is known and can be adjusted for in the pro-

Fig. 3—Flowsheet for the Sterling Process

portioning operation. Course ores having a screen size of 95 pct —½-in. square opening and 95 pct —200 mesh, as well as fine ores of 95 pct —8 mesh and 30 pct —200 mesh, have been smelted successfully. Reducing coal to coke and flux of the same general size ranges have been used without difficulty. As to sizing of raw materials in general, it is important that the mixture does not seriously segregate on preheating and charging, that the dust be held to a minimum, that there be reasonably good contact between ore, coal, and flux particles, and that the angle of repose of the charge banks in the furnaces not be too small.

The chemical requirements of the ore, coal, and flux are likewise not too stringent. The ore must be in the oxidized state with preferably not more than 1 pct total sulphur and/or 1 pct CO_2 present. Ores containing a considerable range of lead, cadmium, and copper have been treated up to the present time. Ores having as high as 20:1 zinc:iron ratio, and as low as 1:1 have been treated successfully. Reduction fuels with 2 to 6 pct volatile matter and 20 pct ash have been used. Fixed carbon preferably should be in the range of 60 to 80 pct.

The ore-fuel-flux mixture is fed, as shown in Fig. 4, into a rotary preheater fired with the CO gas produced as a byproduct of the reduction operation. The time required for the mixture to travel through the preheater is about 2 hr. In this time, the mixture is heated from room temperature to 850° to 900° C, some of the coal volatile is removed, some of the Fe_2O_3 is reduced to FeO, and CuO—if present—is reduced to Cu_2O or Cu. In the preheating, some ore and coal dusts are produced together with some zinc, lead, and cadmium fume. These dusts and fumes are collected in a mechanical bag filter and are recycled unless the percentage of such valuable metallic constituents as cadmium makes it advisable to recover them from this collected material.

The heated mixture as it discharges from the preheater is collected in insulated holding hoppers and from there it is withdrawn in 2 to 3-ton lots at desired intervals to charge to the Sterling furnace, Figs. 5, 6, and 7. The hot charge is conveyed to the furnace by monorail in an insulated car from which the charge is fed directly into a series of charge hoppers located in the roof of the furnace along the inside perimeter of the four walls. The recirculation and charging of byproduct blue powder and dross are carried out in the same manner. The furnace receives a round of charge at 1 hr intervals. The height of the charge banks and the width of the molten bath are checked at regular intervals by rod measurements through probe holes. The distribution

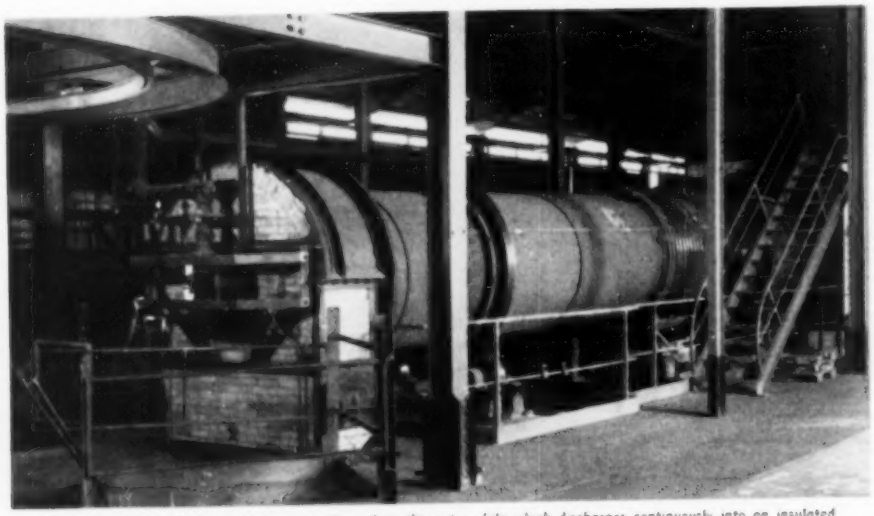

Fig. 4—The Sterling furnace charge is preheated in this rotary kiln which discharges continuously into an insulated hopper on the floor below. The mixture travels through the preheater in about 2 hr.

of the charge is governed by the results of these probe measurements and the amount of charge is dictated by power input.

The molten slag and iron collect in a pool on the hearth of the furnace surrounded by the charge banks. The depth of molten bath carried in the furnace is variable. In general, sufficient iron is carried in the furnace to maintain a protective layer and permit 5 to 10-ton taps; and sufficient slag is maintained over the molten iron bath to avoid any direct contact of the arcs with molten iron. The iron and slag tapping intervals depend on the composition of ore worked. The iron is sand cast and the slag is granulated for further disposition.

The volatile products of the reduction reaction (zinc vapor and CO gas, together with the lead, cadmium, and part of the silver) pass out of the furnace through refractory connectors into condensers of the patented splash type previously described in the literature.[4] The liquid metal collects in the condenser sumps where it is maintained at 500 C by means of thermostatically operated water coils. The bath of molten metal is kept at a relatively constant level in the condenser sumps by continuous overflow or by periodic tapping into a ladle, Fig. 8.

The noncondensable gas (largely CO) passes from each condenser into a scrubber where it is washed with fine water sprays to remove any solid materials. The gas then travels through a water venturi which automatically controls the back pressure on the furnace and also furnishes the motive power to move the clean gas to points of consumption—such as the preheaters and liquating pots. The dried blue powder from the gas scrubber system, together with any blue powder that collects in the condenser, is returned to the furnace.

The molten metal is transferred to a blending and liquating pot where the lead and iron are removed to the extent that the zinc metal may (depending on other impurities in the ore) meet the requirements of Prime Western specification. The iron dross from the liquating pot after granulation or casting is recirculated to the Sterling furnace. The pig lead produced is sent to a lead refinery. Any cadmium in the charge will be contained in the slab zinc metal. Should the market demand a premium grade of zinc, or should the cadmium or other values call for further processing, the molten liquated metal can be refined to produce Special High Grade zinc. In the refining process, the byproduct metal values can be recovered and subjected to further refinement.

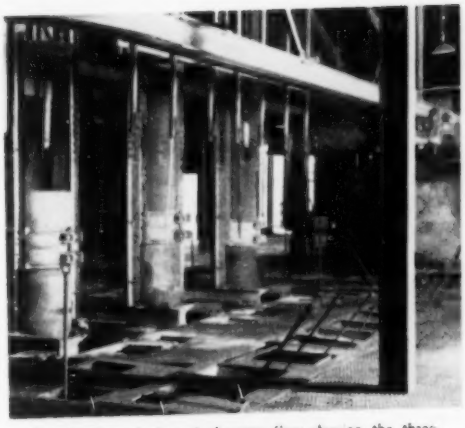

Fig. 5—General view of charging floor showing the three 24 in. diam graphite electrodes and charge hoppers.

Metallurgical Performance

The full sized Sterling furnace has been in continuous operation since July 1951. During that pe-

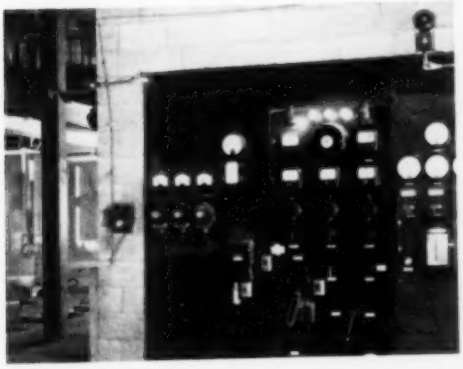

Fig. 6 (Above)—View of the charging floor showing the control panel. The furnace is provided with 6000-kw transformer capacity.

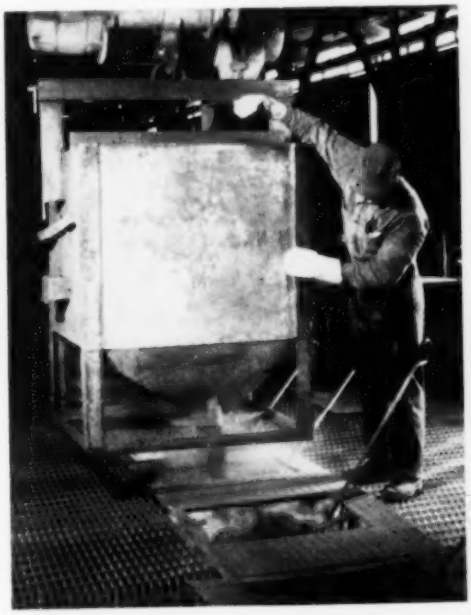

Fig. 7 (Right)—Distributing preheated charge to individual hoppers. The charge is withdrawn in 2 to 3-ton lots at desired intervals to charge to the Sterling furnace.

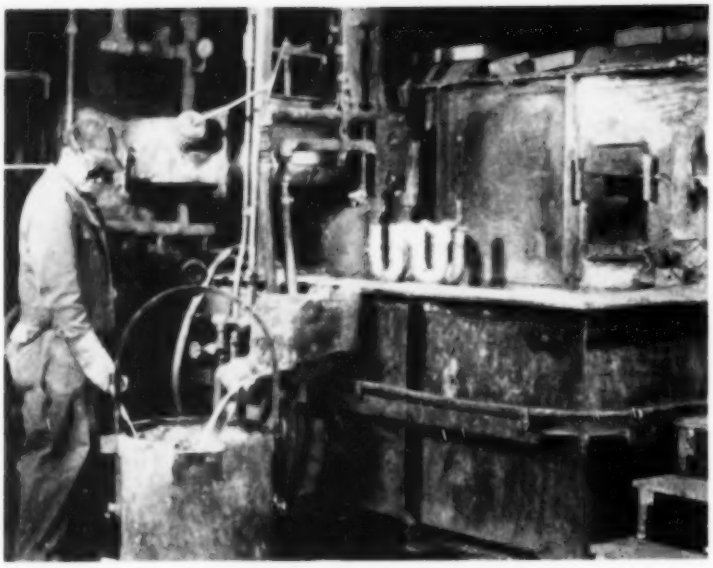

Fig. 8—Molten zinc is being tapped from the condenser reservoir. Gas scrubbers are behind the operator.

cost, runs of a month or more each have been made on several ores and concentrates ranging from Sterling Hill mine crude ore to typical high grade zinc concentrates. The operation was started at a relatively low rate, which has been steadily increased as the operating crew have developed the necessary experience and ability to handle the furnace. The highest sustained rate of power input to date has been 4500 kw, but this is not considered the limit of the furnace capacity. In general, charge and metal handling facilities and condenser capacity have been the bottlenecks encountered, and these limitations have been, or are being, overcome as they appear.

The operating experience has satisfactorily demonstrated that the capacity of the furnace measured in tons of liquated slab zinc—with full recirculation of all condenser and scrubber blue powder, skimmings, and liquating pot dross—is in excess of 35 tons per day when treating a 50 pct or better grade zinc concentrate carrying as much as 15 pct iron. A zinc recovery of 93 pct is obtainable on such a concentrate.

The overall power consumption, including all electric losses, does not exceed 3000 kw-hr per net ton of slab zinc produced. Auxiliary power is estimated at 150 kw-hr per ton. The power factor is 95 pct or better. Operating time (power on the electrodes) on the furnace has been maintained at between 90 and 93 pct.

The electrode consumption has been between 13 and 15 lb per ton of zinc metal. Experience with the refractories at the end of eight months is not yet great enough to afford a basis for estimating furnace life, except to indicate clearly that it will be economically satisfactory. A vital factor in establishing an economical operation is condensation efficiency. With high grade concentrates, it can now be safely predicted that 75 to 80 pct of the zinc vapor entering the condensers will be tapped as molten zinc.

Substantially all of the zinc and secondary values are eliminated from the slag. Pig iron will, in certain cases, yield a substantial credit; any copper and gold present tend to follow the iron, and, under favorable conditions, the iron can be treated in a copper reverberatory or converter or in a lead blast furnace to recover these values. Over 90 pct of the lead and cadmium will be in the molten condensed metal; part of the lead can be recovered by liquation, and the remaining lead and any cadmium can be recovered by refining, if this is economical. Any silver will be distributed approximately two thirds to the zinc and one third to the iron. Silver in the iron can be recovered with the copper and gold by the means previously mentioned, and the silver in the zinc can be concentrated in refining.

The development of the Sterling Process has now reached the point where it has been demonstrated that the difficulties which heretofore have prevented the smelting of zinc in an electric arc furnace have been overcome, and the commercial application of the process may be expected.

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Service Life and Metal Tonnage Increased at Columbia-Geneva Steel Div. and Clairton Works, U. S. Steel Co.

Mixer Linings at Columbia-Geneva Steel Div.

by R. E. Aikens

MOST of the investigations dealing with hot metal mixer linings in the past mainly concerned themselves with the technical aspects of the problem. Determination of the best lining for hot metal mixers cannot be made on a general basis, however. Factors concerning mixer lining life vary considerably in different plants. Among the most important of these conditions are hot metal analysis and temperature, analysis and amount of slag on the hot metal, and temperature at which the mixer is operated.

Numerous tests have been run at the Geneva works involving variations in brick, mortar, and construction in an attempt to improve mixer life. The use of chemically bonded magnesite-chrome brick in sections of the lining subject to the severest erosion has resulted in an increase in service life approximately four times that experienced during the first few years of operation.

From the very first operation until 1950, fireclay brick lined the two 800-ton metal mixers. The original lining was 22½ in. made up of a 4½ in. course next to the insulation and two 9 in. courses. The construction was later changed to two 13½ in. courses for a thickness of 27 in. Severe erosion always took place in a band 8 to 10 ft wide around the entire mixer and in the jambs and the pouring-out spout. The mixers usually had to be taken out of service because jambs or the backwalls became too thin. The mixers have the pouring-in hole on top, 16° off the centerline. Thus, there is little wear caused by the hot metal stream striking the brickwork when poured into the mixer. Bottoms and roofs have never presented any serious problem. Both sections give about five to six years service with only minor patching. The roofs are independently supported on steel shelves.

Many grades and brands of fireclay were used in an attempt to extend lining life. First quality, super duty, and special high fired super duty fireclay were service tested but failed to give satisfactory results. Various types of mortar were used with little noticeable improvement. Several trials were made using burnt chrome-magnesite brick in the jambs and pouring-out spout. Sections of this brick falling off the jambs presented difficulties. Efforts to hold the brick in place by building the jambs with light gage steel plates between courses of basic brick proved unsuccessful. There was little difference in lining life shown throughout the testing program. None gave what could be considered satisfactory service.

The average service life of a mixer lining was 3½ months, with 160,000 tons of hot metal passing through the mixer. Normal practice has been to take the mixer down for repairs when the inside 13½ in. course is completely worn away in any section. Wear usually occurs in the center of the backwall. From the standpoint of economies and mixer availability it has been found to be most advantageous if no brick replacement is necessary in the outside 13½ in. course. The practice of laying the outside course in ring construction and bonding the inside course has been adopted to determine when a section of the inside course is gone. By visual inspection from the pouring-out spout, the difference in construction can be detected and the mixer taken out of service before excessive damage to the outside course occurs.

In June 1950, a section of the inside 13½ in. course approximately 8 ft wide around the mixer, was lined with chemically bonded magnesite-chrome brick, measuring 13½x9x3 in. Each brick was dipped in periclase mortar. The mixer stayed in service 14½ months and had 789,300 tons of hot metal pass through it, for an average of almost 1800 tons per day. Thinning jambs and pouring-out spout forced the mixer out of service. The basic brick section of the backwall was down to 3 in. in the thinnest area. The basic section showed almost none of the usual joint erosion and feathering of brick found when fireclay brick was used. In August 1950, the second mixer was lined in a similar manner. The mixer stayed in service 14 months with 366,000 tons of hot metal through it, for an average of 880 tons per day. The condition of jambs and pouring-out

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spout also caused this mixer to be taken out of service.

The average increase in service life for the two mixers with basic lining was four times that obtained with various types of fireclay brick used, and the average hot metal tonnage increased 3½ times. Both mixers have been relined with the same type magnesite-chrome brick and have been operating five and six months with apparently little wear to the basic area.

The mixers are fired with coke oven gas and the normal roof temperature is held between 2450° and 2500° F. It is felt that the most economic mixer lining life is not as important as furnace production rate. The increase in production rate resulting from high temperature hot metal probably more than compensates for slightly decreased mixer lining life. With the normal charge of 65 to 70 pct hot metal, temperature of the metal is of prime importance. Open top type blast furnace ladles are used for delivering iron to the mixers and appreciable temperature loss occurs during the transfer. Attention is given to getting the hot metal in the mixers as fast as possible.

Burners have been installed for heating the hot metal transfer ladles when they are not in use, to decrease temperature loss in transfer of metal from mixers to the furnaces. Although hot metal temperatures in the mixers cannot be increased by high firing as a regular thing, temperature loss through the mixer can be minimized.

Skimming practice at the blast furnaces is generally good. A typical analysis of blast furnace slag for the last year is: SiO_2, 35 to 37 pct; Al_2O_3, 12 to 14 pct; CaO, 30 to 36 pct; MgO, 15 to 17 pct; S, 1.15 to 1.25 pct; MnO, 0.20 to 0.30 pct; and FeO, 0.30 to 0.50 pct.

The original cost of the magnesite-chrome brick is higher than any of the various fireclay brick used in the past. However, the amount of this basic brick comprises only about 25 pct of the total brick in the main mixer body lining. From the standpoint of mixer repair and availability, the use of this brick has been especially advantageous.

Mixer Linings at Clairton Works

by F. R. Smith

THE intermittent demand for hot metal by open hearth furnaces makes a storage place for blast furnace iron necessary in today's open hearth shops. The importance of the hot metal mixer is most forcibly shown when the mixer is off for repairs, with resulting operating difficulties reflected in noticeably decreased production. Every effort is being made to minimize such outage time by increasing the life of the mixer lining.

During the four years from June 1946 to July 1950 the 1000-ton mixer at the Clairton works of the U. S. Steel Co. averaged 5.7 months between relines, handling an average of 215,000 tons of metal. Approximately 40 pct of the lining was replaced on each repair, and the outage time for these jobs averaged eight days. The lining during this period was of mica schist stone bonded with prepared gunsiter. This 18 in. thick wall was backed with 4½ in. of blast furnace quality fireclay brick as a safety lining. A 12 in. semi-silica roof stood throughout this period.

As is generally the case, the most severe erosion occurred in the joints between the rough mica schist stones. However, the joints were not the only weak points as the stones also eroded quite rapidly.

The mixer is cylindrical with the exception of the front wall, built vertically to meet the spring of the roof. It is approximately 15 ft diam and 34 ft long inside, with a top side pouring-in spout 7½ ft from one end and a side pouring-out spout 8 ft from the other end. The mixer is fired with coke oven gas.

In an effort to prolong the life of the lining, and with an eye toward the success of the Ohio works with this material, it was decided in July 1950 to try a refractory lining of cone 23 super duty brick with silimanite mortar. This brick has a fusion point of cone 33 to 34, corresponding to a temperature of 3173° to 3200° F and is dense and strong.

The safety lining was built of regular fired blast furnace quality brick and was 13½ in. thick on the bottom and up the back wall, to a point approximately 2 ft below the normal metal line, where the thickness was decreased to 9 in. On the front wall the safety lining acted as a filler between the perpendicular super duty wall and the shell of the mixer, and varied in thickness from 9 in. to approximately 15 in.

The super duty lining was 13½ in. thick on the bottom and up the back wall to the point where the safety lining was decreased. Here the thickness increased from 13½ to 18 in. The vertical front wall was constructed 27 in. thick with super duty quality brick. The ends, which had suffered considerable erosion in previous linings, were built to a 27 in. thickness with the super duty brick. Directly under the pouring-in spout, a section 10x10 ft was made 18 in. thick with super duty to ease heavy erosion. The bottom and jambs of the pouring-out spout were constructed of 18 in. super duty block set on end. A 12 in. semi-silica roof was built at this time.

The mixer was in continuous operation from August 1950 to February 1952, with one patch job on the bottom and jambs of the pouring-out spout. No appreciable wear was noted at any point. It passed 820,000 tons of metal in the 18 months, an increase of almost 400 pct over the average tonnage during the preceding four years.

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New Chemical Method Recovers

Nickel Cobalt Copper | Metal

DEVELOPMENT of a chemical process for the extraction of pure metals from mill concentrates or metal scrap has progressed beyond the pilot plant stage and may prove an important adjunct to present smelting and refining methods if initial commercial operations prove economically successful.

Developed by the Chemical Construction Corp., the new process involves the treatment of oxide and sulphide ore concentrates by chemical methods, instead of the usual smelting and refining techniques. Several of the many applications are now scheduled for commercial use. Refiners using the new process will prepare ore concentrates by standard flotation methods, introduce the concentrate as a slurry into an autoclave along with water and an acid or ammonia. From the resulting leach solution, recovery of individual metals is made by use of suitable reducing agents. By varying conditions during treatment, different metals in the ore are produced separately as pure powders, which may be pressed into forms ready to market, or in the case of copper, extruded as rods or pipe. The reagents are generally recovered. By manipulating the variables during reduction, selective separation of nickel, and/or cobalt, and/or copper can be made simultaneously. The separation is a continuous operation.

Low metal prices are not immediately in view because of the tremendous demand at this time. However, reduced metallurgical treatment costs will allow economical mining of orebodies with lower metal content, permitting increased production to meet demand. Although the practical horizon for use of the new process appears to be limitless, one factor may enter into its employment. Just how low grade a concentrate can be economically used is unknown. In addition, for present practical considerations, only metal below zinc in the electromotive series may be processed.

Each commercial application requires specific technique adaptation and pilot-plant data for engineering design. A nickel-cobalt-copper process has been researched and piloted in collaboration with Sherritt Gordon Mines, Ltd., for the firm's Lynn Lake properties. In addition, processes were tailored for cobalt concentrates of Howe Sound Mining Co., and National Lead Co., in view of the urgent need for this specific metal.

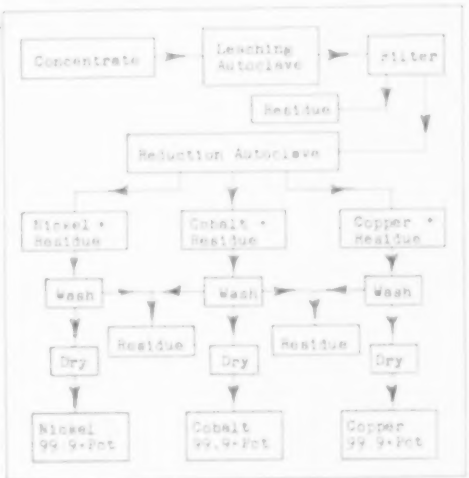
Major General William N. Porter, president of Chemical Construction, says of the new process, "piloting experience has shown that production cost, from ore concentrates to pure metals should be considerably below current costs." Other savings may be realized by cutting transportation and personnel costs, and by reducing the time lag between mining and pure metal from months to a matter of hours.

First commercial use of the process will start sometime this summer. Chemico is slated to finish building a $2.5 million cobalt refinery for Howe Sound Mining near Salt Lake City. The plant is expected to raise world cobalt production by 40 pct. The plant will process 35 tons of 20 pct cobalt concentrates from Howe Sound's Blackbird mine near Cobalt, Idaho, daily. Yearly production is expected to reach 2000 tons of pure metal, about one half of U. S. consumption in 1950.

Steps in the Howe Sound application of the process are:

1. Acid oxidation leach.
2. Filtration (residues thrown away are insoluble compounds—gangue, iron, arsenic).
3. Cementation (for Cu removal because Cu content is too low for recovery).
4. Reduction from ammoniacal solution.
5. Separation of Co and Ni as mixed metals (95 pct Co and 5 pct Ni).
6. Recovery of ammonium sulphate.

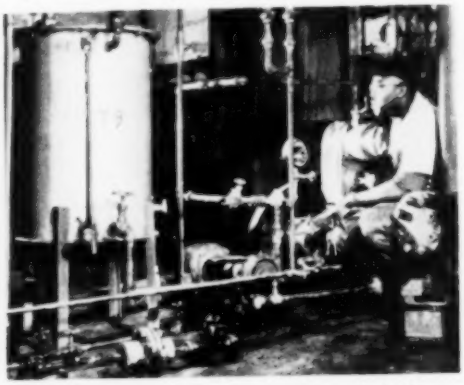
Under construction at the Fredericktown, Mo., mine of National Lead is a $5 million refinery sched-



Hypothetical flow sheet for the simultaneous recovery of nickel, cobalt, and copper. Basic operations are performed in autoclaves, under controlled pressure and temperature.

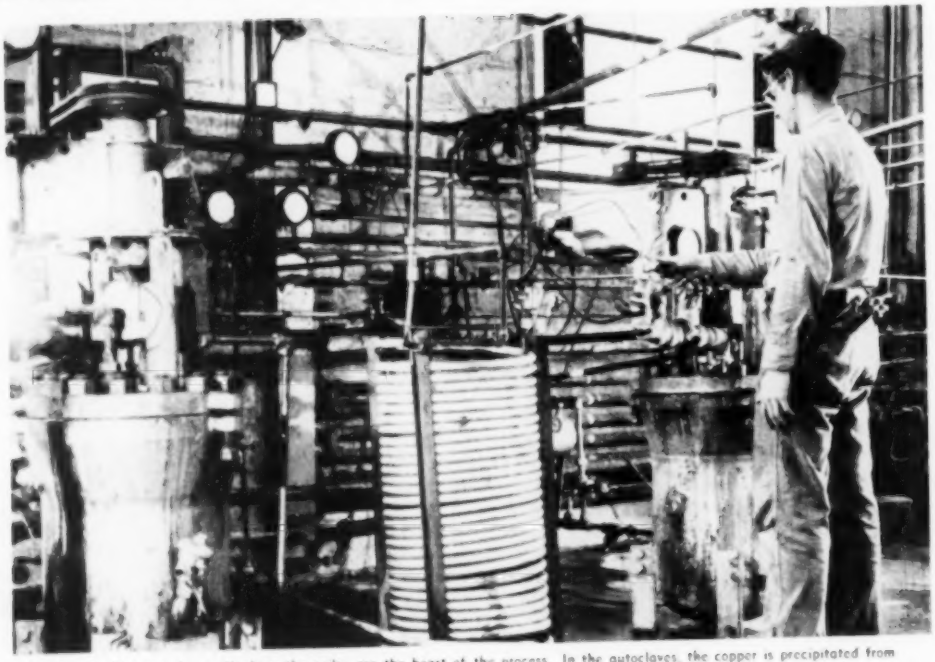
...uled for completion in 1953. It will have a design capacity of 50 tons of concentrate per day. National Lead plans an annual production of 700 tons of cobalt, 900 tons of nickel, and 700 tons of copper, in addition to 7500 tons of fertilizer-grade ammonium sulphate.

Recovery of copper from copper scrap is shown at Chemico's pilot plant in Linden, N. J.



Step 1 (Left)—Basket of scrap is lowered into the leaching solution at the beginning of the process. This, and other steps will be fully mechanized in commercial plant operation. Leaching is done in autoclaves.

Step 2 (Above)—Solids are filtered out of the solution. The solution is then pumped to the measuring tanks feeding the reduction autoclaves.



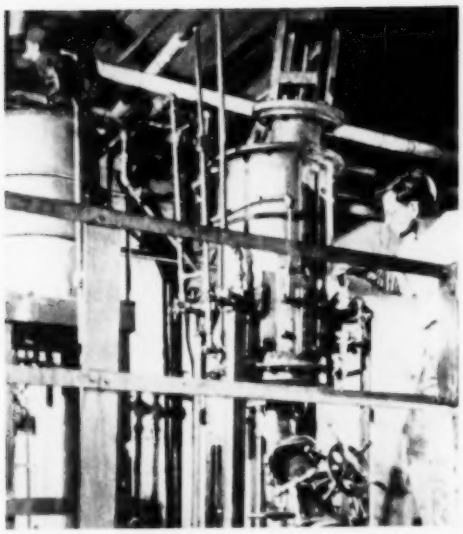
Step 3—The autoclaves (flanking the coils) are the heart of the process. In the autoclaves, the copper is precipitated from solution under high temperatures and pressure with reducing agents. Two coils, one inside the other (center), are for heat exchange—heat from precipitated copper leaving the autoclaves is transferred to solution going into them.

Another development is the $17 million nickel refinery under construction for Sherritt Gordon at Edmonton, Alberta. Sherritt Gordon's ammonia leach process and Chemico's nickel reduction process are to be combined. Annual production is anticipated at about 8500 tons of nickel, 1000 tons of copper, 150 tons of cobalt, and 70,000 tons of ammonium sulphate. The Edmonton plant is scheduled for production in the fall of 1953.

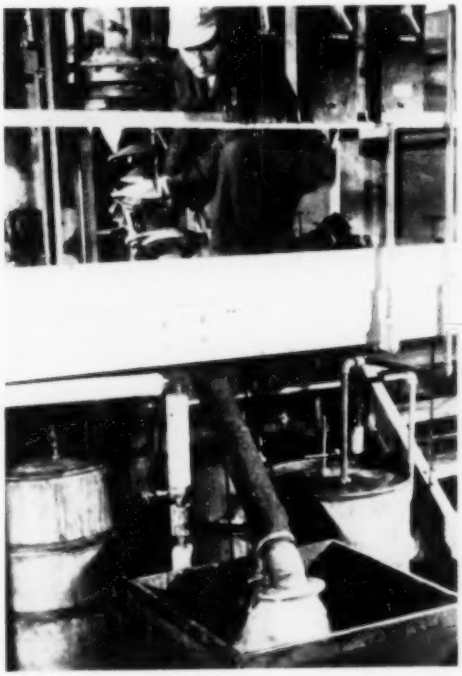
One of the most significant developments from the new process which may soon find commercial application is a method for production of pure copper powder from any form of copper scrap, brass scrap, or blister copper. The process is said to achieve almost 100 pct recovery. The product, better than 99.9 pct pure, meets all standards for oxygen free, high conductivity electrolytic copper. The process was developed in collaboration with Chemetals Corp.

In September 1951, Chemetals began collaboration on a technique to produce sintered manganese oxide from low grade manganese ores. Chemetals and Chemical Construction will start joint pilot operations in the near future. A moderate amount of material is needed to make the changeover from smelting and refining methods to the new process, according to General Porter. Efficient and compact plants may be built at the mine site with accompanying cuts in transportation costs. Other advantages pointed out for the process are the overcoming of inventories of partially processed material piled up because of distance between smelters and refiners. Present time lag in production may extend over a period of months.

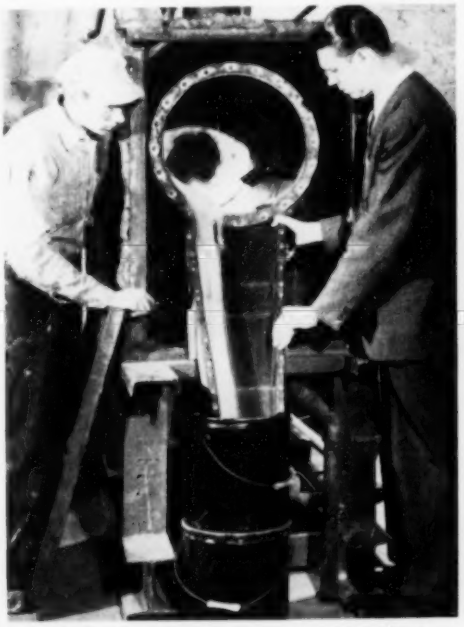
Rights to many of the processes are shared by Chemical Construction and Sherritt Gordon, the latter building and operating a pilot plant at Ottawa. Rights to the copper scrap and manganese processes are shared by Chemical Construction and Chemetals.



Step 4—Washing tanks receive the copper after it is precipitated in autoclaves and wash away leaching solution



Step 5—Water and powdered copper pour out of washing tank into box filter for preliminary drying.



Step 6—Gleaming copper powder pours from rotary drier in the process's last step. George Van Hare, right, of Chemical Construction inspects the powder. Equipment for continuous drying will be installed in commercial plants.

Effect on Production Rates of

Foreign Ores as Open Hearth Charge Ore

Liberian Ore

by R. P. Carpenter

FACED with a lack of good charge and feed ores, Republic Steel Corp. started a program in 1949 which eventually led to the import of what experts have termed the finest mined ore in the world. In June 1951 the Cleveland plant received the first ore cargo taken from new deposits near Monrovia, capital of Liberia. The physical and chemical characteristics of available ore has been a controversial subject for many years relative to a particular ore's effect on open hearth production. In the case of high hot metal charges, the ore determines to a great extent the success or failure of the operation.

Sinter ore as charge was standard practice in the 15-furnace shop, tapping 200 tons heats, on a 63 pct hot metal charge. The characteristics of this magnetite sinter resulted in several operating problems. The use of sinter, particularly in high metal charges, appeared to be no help in obtaining a high production rate, because of the great amount of fines resulting from low bulk density. In addition, the sinter contains a high percentage of extraneous elements not conducive to substantial production rates.

Heats charged with this type of sintered ore developed a foaming action that did not permit fast

Table I. Ore Analysis, Pct

Iron	68.47
Silica	0.73
Alumina	0.75
Lime	0.09
Magnesia	0.07
Manganese	0.06
Sulphur	0.015
Phosphorus	0.040
Moisture	
Loss on Ignition	0.63

flushes. As a result, the time factor between metal addition to first feed ore addition increased excessively, with a lengthening of heat time. The use of this sinter was detrimental to furnace roof refractories. The ore floated into the slag and melted in the later stages of the heat, when the roof was operating at maximum temperature. Iron oxide fumes permeated the furnace atmosphere to the extent that the operator was unable to see the roof even with the fuel off. In addition to shortening roof life, the excessive blowing resulted in serious floor spills. Production rates, during the first quarter of 1951, averaged 15.0 tons per hr.

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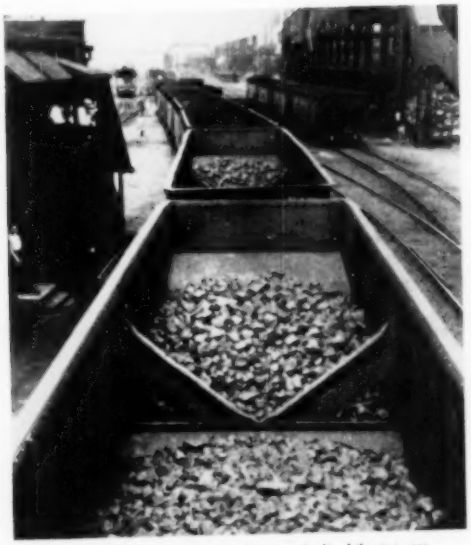

Fig. 1—The ideal size distribution of the Liberian ore received can be seen in this photograph. The ore consists of hard, dense lumps, and is free of fines.

Chemical analyses of 50 cars of the first Liberian shipment showed 68.47 pct Fe content, (Table I) desirable since it was associated with high available oxygen. Silica and alumina acid oxides were present but in minute quantities. Moderate amounts of basic oxides such as lime, magnesia, and manganese oxides were not excessive. The sulphur content was extremely low. Because the basic open hearth is an efficient eliminator of phosphorus, its presence in the ore cannot be considered significant. Although moisture is present in both natural and combined states of the Liberian ore, it offers no serious problem and does not contribute to violent blowing. Fig. 1 shows the physical content of the ore consists of hard, dense, lumps free of fines, with size distribution ideal.

The operator is concerned with chemical reactions in the furnace when hot metal and charge ore react, only to the extent of the effect on subsequent melting and refining results. Liberian ore reacts quickly with hot metal with a controlled and uniform action.

The silicon, manganese, and phosphorus in the hot metal oxidize rapidly, forming a hot fluid slag, free of foam, which runs quickly out of the flush hole. The ore has no tendency to float in the slag or to be carried out of the furnace with the flush. The use of the same ore as a feed ore also speeds up carbon reduction and is three times as effective as previously used feed ores.

Heat charged with the Liberian ore took metal with less reaction, minimizing slag spills and floor congestion. Tons per hour operating performance shows an increase of 1 to 1.5 tons per hr for the shop using heats charged with the imported ore. Charge ore, feed ore, and limestone consumption were considerably less.

It is believed that the Liberian ore more closely approaches the ideal charge and feed ore than any other used in the industry today. Liberian ore outperformed Brazilian ore tested previously. It produced the highest production rates and yields, the lowest bottom and bank delays, the best melt carbon control, and higher average roof life, using the least amount of ore, limestone, and fuel. Finally, the first shipment was used without practical limits on the percentage of iron in the charge in fear of blowing or spills.

Moroccan Ore

by C. C. Benton

FAMILIARIZATION with differences in shop conditions throughout the industry is necessary for evaluation of results obtained from the use of any particular ore for charge oxide, which in the case considered here is Moroccan ore. The average hot metal analysis at the Fairchild steel works in 1951, prior to the use of import ore, was 0.85 pct silicon, 0.25 pct manganese, 0.81 pct phosphorus, and 0.048 pct sulphur. Because of this analysis, 235 to 240 lb of limestone per ton of ingots were charged.

Approximately 30 pct of all heats were made with flush slag practice. Limestone reduction is impossible with flush slag practice and thus a minimum of 60 pct hot metal is required to obtain flush. Seventy percent of all heats tapped are maximum 0.10 pct carbon, with the majority 0.08 pct maximum carbon.

Moroccan ore first was used as a charge at Fairfield in April 1951. From April 1951 through January 1952, 2054 heats ore were tapped. The size of Moroccan ore is good with 26 pct above 3 in., 41 pct from 1 to 3 in. and only 10 pct below 0.25 in. The moisture content is approximately 2.7 pct. Physical characteristics of Moroccan ore are compared with those of Brazilian ore in Table I.

The reaction on metal addition is vigorous with quick rise and fast flush. Flush begins from 5 to 13 min after metal addition and finishes in approximately 30 min. No foaming or excessive blowing has been experienced.

Iron loss is greater than that of Brazilian ore but less than occurs with iron ore sinter. The ingot yield on Moroccan ore has been 87.22 pct.

The chemical analysis of Moroccan ore is good except for the high sulphur content. Sulphur pickup from the ore charged averaged 0.005 pct with no difficulty in meeting sulphur specification except when using a high sulphur fuel for melting. Chemical analyses of Moroccan and Brazilian ores and iron sinter are given in Table II.

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Table I. Screen Analysis of Moroccan Ore, Pct

Screen Size	Moroccan Ore	Brazilian Ore
+3 in.	26.1	30.3
3 × 2½ in.	16.0	2.7
2½ × 1½ in.	20.9	4.9
1½ × 1 in.	10.4	8.7
1 × ⅜ in.	11.2	8.5
⅜ × ¼ in.	11.4	8.6
¼ × 3/16 in.	1.7	11.5
3/16 × 10 mesh	2.2	2.8
10 × 20 mesh	1.3	5.8
20 × 40 mesh	1.6	4.5
40 × 60 mesh	0.8	0.1
60 × 80 mesh	0.5	5.8
80 × 100 mesh	0.2	1.2
100 mesh	1.7	4.6
	100.0	100.0

Table II. Chemical Analysis of Moroccan Ore, Pct

	Moroccan Ore	Iron Sinter	Brazilian Ore
Metallic iron	63.74	67.93	68.49
Silica	3.53	1.98	0.43
Alumina	0.67	0.17	0.51
Lime	0.98		
Magnesium	0.44		
Manganese	0.23	0.04	0.07
Sulphur	0.226	0.093	
Phosphorus	0.028	0.006	0.036
Copper	0.036	0.111	

Table III. Comparison of Heats Using Moroccan Ore with Other Charge Oxides

	Moroccan Ore	Iron Sinter	Brazilian Ore
Tons per heat	189.77	190.13	190.50
Time of heats, tap to tap	10.41	10.59	10.33
Time of heats, charge to tap	9.29	9.47	9.21
Tap per tap, tons per hr	17.86	17.31	18.06
Charge per tap, tons per hr	20.02	19.44	20.38
Charge oxide per ton ingots, lb	177	188	160
Feed oxide per ton ingots, lb	54	67	60

From studies on use of various charge ores, considerable sulphur is removed on flush. Results of the heats show Moroccan ore can be used in combination with low sulphur fuels with advantage to production. Table III compares heats using Moroccan ore and heats using other charge oxides.

MPA Holds Eighth Annual Meeting in Chicago

High Temperature Materials, Parts Production, and Military Requirements for Powder Metal Parts Stressed in Technical Sessions

ALMOST 600 powder metallurgists, parts producers, and equipment suppliers registered for the eighth annual meeting and 1952 Metal Powder Show of the Metal Powder Assn., held in Chicago, Apr. 29 and 30, 1952. Some 28 manufacturing firms exhibited their wares during the show, and much know-how was exchanged outside meeting rooms.

Technical sessions were held Tuesday morning and afternoon, and Wednesday morning. Tuesday evening was devoted to the reception and buffet supper, and a luncheon was held Wednesday.

T. L. Robinson, the Wel-Met Co., served as chairman of the first session. A paper on *Recent Practical Applications of Hot Pressing*, was presented by J. F. Kuzmick, Welded Carbide Tool Co. He gave an overall picture of hot pressing methods, equipment, and applications. Induction and resistance heating methods were covered. One aspect discussed was a means of salvaging carbide bearings when the ID became worn. Usually the bearings are discarded, or else reground to the next largest bearing size, an expensive and time consuming procedure. These worn bearings were successfully recompressed, ending up with the original ID, the height is shortened in the process, but that is usually not a critical dimension. Mr. Kuzmick also dwelt on diamond bits for rock drilling, carbide parts and dies, and heavy metals. He opined that hot pressing could become a full scale production method similar to the present cold pressing and sintering, although no full scale production is in existence today. One drawback is that no real satisfactory mold material has yet been discovered, and most molds, usually carbon, can only be used once, adding to the cost of the process.

The use of powder metal parts in large scale and precision production was described by Fred Lux, Lux Clock Mfg. Co., in his talk on *Powder Metallurgy in the Manufacture of Clocks and Mechanisms*. Using carbide dies, powder metal gears, cams, wheels, etc., are produced accurately and inexpensively. Mr. Lux pointed out that the inexpensive clock has a useful life of 10 years, although average clock life is 2 to 3 years because of mishandling.

The afternoon session had as chairman E. H. Klein, N. J. Zinc Sales Co. The lead off paper, *Aspects of Powder Metallurgy in National Defense* was given by Capt. John D. Dale, Ordnance Corps, Frankford Arsenal. He pointed out that development work was started on well over 100 new components in the past year alone. Those components already in production have saved the taxpayer over $2 million, and those that are expected to be in production before the end of the year will save an additional $10 million. In the event that sintered iron rotating bands and small arms ammunition are made, an additional saving of $8 million per year would be realized.

He went on to discuss the manufacture of components for mechanical time fuses, VT fuses, caliber .50 cartridge cases of sintered iron, and a 20 mm projectile of sintered iron.

Capt. Dale discussed at length the problems involved in adopting the sintered iron rotating band, which had been much ballyhooed as being ready for production. He pointed out that the rotating band

was only one element of a shell or projectile, and that projectile had to be perfect in order to be accurate. The many mechanical, cleaning, and finishing operations experienced by the assembled band, together with the terrific forces exerted during the firing period, resulted in erratic results during early firing tests. Sintered rotating bands will quite possibly be produced as soon as design aspects, type of impregnating material, surface preservation methods, etc. have been thoroughly worked out. To insure maximum strength in the rotating band blank, the iron powder that will be used will be one that sinters at the lowest temperature in the shortest time and permits complete recrystallization across original particle boundaries.

Julius J. Harwood, Office of Naval Research, presented a timely and informative paper on *Powder Metallurgy in High Temperature Applications*. His topic included conservation of critical materials and the need for new high temperature materials. He pointed out the requirements that must be met in jet engine service, and the applicability of powder metal parts. Whereas forged stainless steel blades were once used exclusively in the compressor, at least one commercial engine now employs powder metal blades as standard, and the blades are being tested for rotor applications. These blades were developed by American Electro Metal Corp., and are in production by Thompson Products Inc. Titanium and titanium alloy development is proceeding on a large scale, and Westinghouse is using a titanium alloy as compressor disc material. Since titanium powder can now be pressed to the same density and properties as are melted material, he felt that powder metal methods should be able to produce titanium ingots from sponge that would be superior to and less expensive than the cast material. Also coming up for mention were titanium carbide compositions, which show superior high temperature properties, sweat cooling, and parts such as vanes and nozzles that must withstand extreme temperature variations.

Malcolm F. Judkins, Firth Sterling Steel & Carbide Corp. spoke on *Carbides-Machining and Ordnance Applications*. He outlined various aspects of carbide design, and spoke about the Method X method for machining hard metals.

Wednesday morning, under the chairmanship of W. E. Cairnes, Radio Cores, Inc., a symposium was held on Electronic Core Materials. G. O. Altman, General Aniline & Film Corp. spoke on carbonyl iron powders; R. H. Rodrian, C. K. Williams & Co., spoke on hydrogen reduced iron powder, and W. M. Shafer, Plastic Metals Div. National Radiator Co. presented data on electrolytic iron powder.

Eberhard Both, Signal Corps Engineering Laboratories followed the symposium with a very practical paper on *Magnetic Powder Cores for Military Communication Equipment*, in which he described military needs and applications and results of tests conducted on cores under various operating conditions met with military communications equipment.

One of the world's foremost powder metallurgists, W. D. Jones, Powder Metallurgy, Ltd., London, the largest powder metal company outside the U. S., presented the final paper of the session, *Progress in Powder Metallurgy in Europe*. This account, a detailed description of the industry, country by country, was compiled by Dr. Jones as a result of personal visits. He said that Sweden was producing the best rock drills, and that Phillips in Holland had a staff of 4000 research and development personnel. Phillips has developed an emission microscope that makes possible a study of the changes in powder metal parts at high temperatures. Countries covered in the talk were Sweden, Holland, Germany, Belgium, France, Austria, Spain, Portugal, Italy, England, and South Africa. In Germany, sintered magnets are being produced at the rate of 20 tons per month in batch operated vacuum sintering furnaces. Metalwork Plansee in Austria was recommended as one of the finest and most progressive plants in the world. South Africa has one of the most modern carbide plants to be found anywhere. Carbide consumption for rock drills in Africa is 10,000 tons per month and increasing.

At the closing luncheon, Russell E. Settle, Allison Div. General Motors Corp. presented an interesting talk on jet engine production, problems, supply, and achievements. The next annual meeting of the MPA and 1953 Metal Powder Show will be held in Cleveland, Apr. 20 to 22, 1953.

Delegates to eighth annual Metal Powder Assn. meeting are shown crowding the exhibit section in the pictures on the left and right. Almost 600 metallurgists, parts producers, and equipment suppliers registered for the event. Some 28 firms occupied the exhibit booths, taking part in a constant exchange of information with conventionites. Technical sessions, and informal gatherings gave delegates a busy time during the two-day session. Army Ordnance men and world famous metallurgists participated in the technical sessions, presenting a wide scope of information.

Recrystallization of Tantalum

by G. W. Wensch, K. B. Bruckart, and R. H. Deibler

PRIMARY recrystallization kinetics of high purity tantalum (99.9 pct), which had been reduced 40 pct in thickness by cold rolling, were studied and the kinetic relations determined. Tantalum strips, 6x0.5x0.075 in., were isothermally annealed in a vacuum at 0.5 micron Hg with the strips as the heating elements. Temperature gradients throughout the specimen length were largely eliminated by decreasing the cross-sectional area of the specimens at the cold electrode clamps.

Temperature sightings were taken normal to the flat surface of the specimens at midcenter, using a Leeds & Northrup optical pyrometer. Sighting was through a glass window of approximately 95 pct transmission, and black body temperature corrections were made using published emissivity values.[1]

Each specimen was annealed at constant temperature by controlling the energy input so the brightness of the specimen remained constant by comparison with a predetermined setting of the optical pyrometer. Time at temperature was measured to 0.1 min with a stop watch. Heating times to the annealing temperatures were short as a modified pulse annealing procedure was used.[2] A larger current was used initially to bring the specimen to temperature after which a lower current was used to maintain the annealing temperature. Recrystallization effects occurring during the cooling cycle appear negligible as determined by the method of kinetic analysis of Parkins, Dienes, and Brown.[3]

The approximate times necessary for complete recrystallization were found by microscopic examination of representative electropolished and etched surfaces. After a time bracket was established roughly for each temperature, additional specimens were annealed at a specific temperature using a reiteration change in annealing time until a reasonably accurate time bracket was established.

Table I. Time Temperature Data for the Recrystallization of 40 Pct Cold Rolled Tantalum

Annealing Temperature, °K	Annealing Time, Min	State of Tantalum*
1520	110.00	Partial recrystallization
1520	140.0	Full recrystallization
1570	50.0	Partial recrystallization
1570	55.0	Full recrystallization
1620	12.0	Partial recrystallization
1620	14.0	Full recrystallization
1670	3.0	Partial recrystallization
1670	3.5	Full recrystallization

* Photomicrographs, grain size 0.040 mm diam.

The recrystallization state of the specimens, which were used in finally placing the end point of recrystallization, was determined by X-ray back reflection patterns. Copper K-alpha radiation was used to examine the 321 family of planes. The presence of the resolved doublet was used as the criterion for the existence of unrecrystallized material. The intensity integration of the 48 contributing planes of this (hkl) family permitted a thorough statistical survey of the recrystallization state of the tantalum lattice.

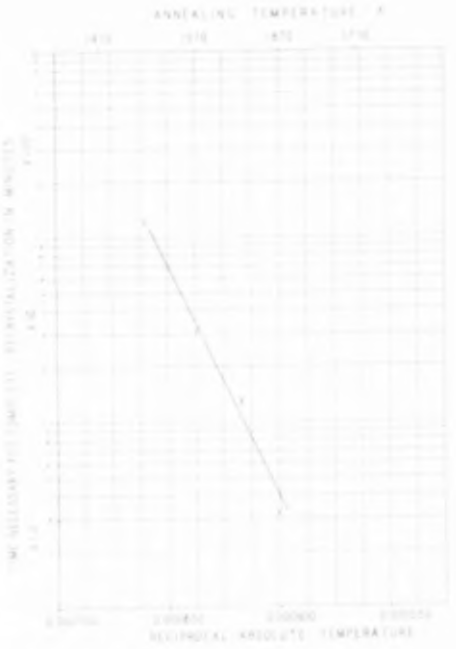

Fig. 1—Plot of time temperature data given in Table I for the recrystallization of 40 pct cold rolled tantalum

Experimental data are given in Table I and plotted in Fig. 1. The heat of activation for recrystallization was found to be 124 ±4 kcal per mol—considerably higher than the values reported for other metals.

Acknowledgment

The authors express their gratitude for the assistance given by Dr. L. F. Yntema, director of research, and R. W. Yancey, chief metallurgist of the Fansteel Metallurgical Corp.

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Activity of Sulphur in Liquid Iron and Steel

by Charles W. Sherman and John Chipman

IN the mathematical statement of the law of mass action, the activity of each substance consumed or produced in a reaction is used to obtain a numerical constant which is characteristic of the equilibrium of that reaction at the given temperature. The activity in many cases is equal to, or proportional to, the concentration. If this is the case, any units denoting concentrations, such as weight percentage or mol fraction, may be substituted in the equilibrium expression. It is even permissible to use mixed units provided only that each unit is that used in defining the activity of the corresponding substance.

The indiscriminate use of concentration units in writing equilibrium constants sometimes leads to very erroneous results. On the other hand, what appear to be contradictory results from similar investigations often might be resolved if the activity is the quantity that is used to determine the equilibrium constant for the reaction in question. Desulphurization serves as an important illustration.

Sulphur in Iron and Steel

The control of the sulphur content of the finished product is one of the oldest and broadest problems in the metallurgy of iron and steel. In general, this control must be exercised through the action of a slag, and for this reason experimental studies of the distribution of sulphur between slag and metal have assumed considerable importance. Under slags of the type found in the basic open-hearth furnace the ratio (% S)/[% S] does not in general exceed about 8. Under blast-furnace conditions, however, desulphurization ratios of 50 to 100 are readily obtained, and under laboratory conditions using similar slags, the ratio may be as high as 400.

The fact that blast-furnace metal is much more readily desulphurized than is molten steel rests upon two distinct phenomena. The first of these is connected with the slag reaction. In the open-hearth furnace, the equilibrium in the following reaction places a definite limit on the sulphur content of the slag:

$$\underline{S} + O^{=} = \underline{O} + S^{=} \qquad [1]$$

where the symbols underlined indicate elements dissolved in the liquid metal, while slag constituents are represented as ions. The high oxygen content of the open-hearth bath prevents this reaction from proceeding far enough to effect good desulphurization. In the blast-furnace process, however, the oxygen activity is limited by the presence of carbon in the metal and in the coke bed and the controlling reaction becomes:

$$\underline{S} + \underline{O} + \underline{C} = \underline{S} + CO(gas) \qquad [2]$$

The second phenomenon which contributes to the easier desulphurization of blast-furnace metal is the effect of high concentrations of other elements. These elements, notably carbon, silicon, and phosphorus, increase the thermodynamic activity of sulphur several fold. As a result of this increase, the escaping tendency of sulphur from the metal phase is increased and its removal facilitated.

Previous Studies

It becomes, therefore, a matter of importance to determine the effects of various elements upon the activity of sulphur in the melt. The experimental approach to this problem has been made through the equilibrium between sulphur in the melt and gaseous atmospheres containing hydrogen and hydrogen sulphide. The reaction and its equilibrium constant are written as follows:

$$\underline{S} + H_2(gas) = H_2S(gas); K_1 = \frac{p_{H_2S}}{p_{H_2} \cdot a_S} \qquad [3]$$

where the symbol a_S represents the activity of the sulphur in solution in the metal.

The understanding of the chemical behavior of sulphur in steel and pig iron rests on a firm knowledge of the binary system Fe-S. A number of studies of the equilibrium have given closely agreeing results from which the activity and free energy of the dissolved sulphur may be found. The recent paper by Sherman, Elvander, and Chipman[1] includes a discussion and quantitative comparison of the results of several of the significant researches[2-7] on dilute solutions of sulphur in iron.

The results of this study indicate that the relationship between the ratio p_{H_2S}/p_{H_2} and the percentage of sulphur in solution is not a linear one. This

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Discussion on this paper, TP 3302C, may be sent, 2 copies, to AIME. Manuscript, Nov. 28, 1951. Los Angeles Meeting, February 1953.

Fig. 1—Effect of sulphur concentration on [illegible] and [illegible]

Fig. 2—Effect of manganese on activity coefficient of sulphur

means that the activity of sulphur is not exactly equal to its concentration, and hence, that the quantity

$$K'_{[\%S]} = \frac{p_{H_2S}}{p_{H_2}\,[\%\,S]} \qquad [4]$$

is not a true constant. Its value at 1600 C was found to shift with sulphur content in the manner shown in Fig. 1, where it is evident that log $K'_{[\%S]}$ is approximately a linear function of the concentration of sulphur expressed as its percentage by weight in the melt. The true value of $K'_{[\%S]}$ is determined from the intercept of the line at zero concentration. This procedure utilizes the infinitely dilute solution as the reference state, making $a_{[\%S]} = [\%\,S]$ at zero concentration. The ratio of $K'_{[\%S]}$ to the true $K_{[\%S]}$ gives the activity coefficient of sulphur defined by the equation

$$f_{[\%S]} = a_{[\%S]}/[\%\,S] \qquad [5]$$

Since log $K'_{[\%S]}$ is linear with the percentage of sulphur, the same is true of log $f_{[\%S]}$. This is shown in the lower line of Fig. 1 where it is designated log $f'_{[\%S]}$, the prime referring to the binary system Fe-S as distinct from the ternary systems to be discussed later.

Three other investigations yield comparable values of $K'_{[\%S]}$ from which activity coefficients may be determined. The results of Morris and Williams[6] and of Morris and Bucld are included in Fig. 1. The two observations of the latter have been corrected for the small amount of aluminum present by a method which will be obvious from the subsequent discussion of ternary systems. The line representing the data at 1615 C is drawn parallel to the 1600 C line and higher by the amount required by the small temperature coefficient of the reaction. The agreement among the three sets of data is so good as to leave little doubt of the correctness of the slope and of the activity coefficients thus determined. The data of Rosenqvist and Cox,[7] in which the agreement is less satisfactory, will be discussed later in connection with the effect of copper.

Experimental Method

The essential features of the experimental method described in the previous paper[1] have been retained in the present work. A prepared mixture of hydrogen and hydrogen sulphide of constant composition was conducted over molten iron containing various alloying elements until equilibrium was reached at 1600 C. The melt was sampled during a run by dipping a silica tube into the melt through an opening in the top of the furnace and drawing up a sample. These furnace samples were analyzed for sulphur, using the entire piece, to determine when equilibrium had been reached. The final amounts of sulphur and other alloying elements were determined from an analysis of the melt after solidification.

Temperatures were measured by sighting a disappearing-filament optical pyrometer on the melt. The instrument was checked periodically against the melting point of electrolytic iron in hydrogen, 1535 C. The true temperature of pure iron was determined from the work of Dastur and Gokcen,[8] which was done under similar experimental conditions. Heats containing large amounts of alloying material were checked by comparison of the optical temperature with that of a tungsten-molybdenum thermocouple. No significant changes in emissivity were detected as a result of adding alloying elements and the temperature corrections for pure iron were applied to all alloys.

It was necessary to prepare the melts in such a manner as to prevent the erosion of the walls of the alumina crucible by the sulphur and sulphides in the charge. For each heat an iron crucible weigh-

Table I. Equilibrium Data on Fe-S-Mn Alloys at 1600 C

Run No.	$p_{H_2S}/p_{H_2} \times 10^4$ (corr.)	Metal Analysis, Wt Pct S	Mn	log $K'_{[\%S]}$	log f_S	log $f'_{[\%S]}$	log $f_S/f'_{[\%S]}$
M-1	4.60	2.11	0.90	2.662	−0.065	−0.059	−0.026
M-2	4.59	2.06	0.60	2.662	−0.073	−0.057	−0.016
M-3	4.42	2.09	1.42	2.670	−0.066	−0.056	−0.010
M-4	2.64	1.25	1.44	2.644	−0.067	−0.035	−0.032
M-5	2.65	1.19	1.97	2.651	−0.074	−0.033	−0.041
M-6	2.64	1.23	2.17	2.668	−0.091	−0.034	−0.057
M-7	2.21	0.99	1.96	2.653	−0.074	−0.028	−0.046
M-8	2.20	1.01	2.04	2.662	−0.085	−0.028	−0.057
M-9	1.93	0.84	1.45	2.639	−0.062	−0.023	−0.039
M-10	1.93	0.83	2.02	2.655	−0.078	−0.023	−0.055
M-11	1.92	0.86	2.61	2.645	−0.068	−0.022	−0.046
M-12	1.70	0.69	2.03	2.667	−0.030	−0.019	−0.011
M-13	1.67	0.76	2.71	2.651	−0.074	−0.021	−0.053
M-14	1.66	0.71	1.30	2.611	−0.034	−0.020	−0.014
M-15	1.52	0.62	2.04	2.611	−0.034	−0.017	−0.017
M-16	1.51	0.64	1.69	2.627	−0.050	−0.016	−0.02
M-17	1.47	0.61	2.21	2.660	−0.083	−0.019	−0.064
M-18	1.44	0.63	1.61	2.641	−0.064	−0.018	−0.046
M-19	1.27	0.59	3.08	2.663	−0.091	−0.016	−0.075
M-20	1.21	0.55	3.49	2.680	−0.101	−0.016	−0.085
M-21	1.16	0.49	2.27	2.648	−0.071	−0.014	−0.057
M-22	1.02	0.46	0.71	2.581	−0.016	−0.011	−0.005
M-23	1.01	0.43	0.91	2.570	−0.043	−0.011	−0.022
M-24	0.96	0.38	0.52	2.580	−0.021	−0.011	−0.010
M-25	0.86	0.35	0.79	2.616	−0.051	−0.010	−0.023
M-26	0.84	0.43	0.61	2.685	−0.108	−0.011	−0.097
M-27	0.83	0.42	4.85	2.706	−0.129	−0.012	−0.117
M-28	0.75	0.39	5.12	2.717	−0.140	−0.011	−0.129
M-29	0.55	0.25	6.30	2.740	−0.175	−0.009	−0.164
M-30	0.54	0.24	6.11	2.732	−0.153	−0.005	−0.147
M-31	0.53	0.23	4.31	2.677	−0.100	−0.007	−0.093
M-32	0.46	0.23	3.47	2.708	−0.211	−0.008	−0.203
M-33	0.43	0.23	0.99	2.581	−0.104	−0.007	−0.177
M-34	0.43	0.23	7.90	2.736	−0.209	−0.007	−0.202
M-35	0.43	0.20	7.96	2.805	−0.228	−0.007	−0.221
M-36	0.39	0.25	8.20	2.800	−0.229	−0.007	−0.222

ing about 150 g was machined from high grade magnet iron. The iron crucible was designed to fit inside the alumina crucible with sufficient space to allow for expansion during heating. The alloying materials were placed in the iron crucible after being mixed with iron powder. Upon heating, the alloy melted first but did not come in contact with the alumina crucible. When the iron crucible melted, the sulphur-rich material was in solution and did not attack the alumina appreciably.

The ratio p_{H_2S}/p_{H_2} was corrected by using the known thermodynamic data for the formation of S_2 which represents the only important dissociation of H_2S at the temperature of 1600°C. Using the corrected ratios and the percentage of sulphur in the metal, it is possible to determine for each heat a value of K', defined in eq 4. The ratio K'/K gives the activity coefficient of sulphur, f_s.

Activity in Ternary Systems

The treatment of ternary systems previously used by one of the authors[5] is suitable for examining the effect of a third element upon the activity coefficient of sulphur. In a ternary solution let f_s represent the activity coefficient of sulphur; also let f_s° be that in the binary Fe-S solution of equal sulphur concentration. The effect of a third element may be represented by the ratio of the two which we shall call f_s^m defined so that:

$$f_s = f_s^\circ \times f_s^m \qquad [6]$$

This method of calculation was successfully applied[5] to the data of Morris and Williams.[4] It has not yet been shown, however, to what extent the value of f_s^m is independent of the concentration of sulphur. The general usefulness of eq 6 will be enhanced if it is found that f_s^m is a function of the concentration of the third component only, and the data to be presented will be examined to ascertain whether this is true.

Fe-S-Mn—Thirty-six experiments were carried out at 1600°C in which the third element, manganese, was present in amounts up to 8.2 pct. The data are shown in Table I. The sulphur content was varied from 0.25 to 2.1 pct, the higher concentrations being limited to the low manganese alloys by formation of a separate phase containing MnS. Values of $\log f_s^{Mn}$ (eq 6) are plotted in Fig. 2. Here different symbols are used to designate heats containing more than 1 pct or less than 0.4 pct or intermediate amounts of sulphur. The three groups of points conform to the line drawn in the range 0 to 5 pct Mn; above this range, only low sulphur points are available. It may be concluded that up to and possibly beyond 5 pct Mn, the coefficient f_s^{Mn} is independent of the sulphur content. It is observed that $\log f_s^{Mn}$ is a linear function of manganese concentration up to about this limit. At higher concentrations, these two empirical observations must be regarded only as useful approximations.

Table II. Equilibrium Data on Fe-S-P Alloys at 1600°C

Run No.	P_{H_2S}/P_{H_2} x 10³ (corr.)	Metal Analysis, Wt Pct S	P	log K'	log f_S	log f_S°	log f_S^P
F-1	0.609	0.231	1.43	2.519	0.058	0.006	0.064
F-2	0.561	0.157	3.21	2.447	0.130	−0.004	0.134
F-3	0.885	0.312	0.76	2.547	0.030	0.009	0.039
F-4	0.717	0.219	2.27	2.485	0.092	0.006	0.098
F-5	0.526	0.133	4.19	2.403	0.174	0.004	0.178
F-6	0.752	0.160	5.69	2.328	0.249	0.004	0.253
F-7	1.078	0.199	6.81	2.266	0.311	0.006	0.317
F-8	0.916	0.149	9.04	2.208	0.369	0.004	0.373

Table III. Equilibrium Data on Fe-S-Al Alloys at 1600°C

Run No.	P_{H_2S}/P_{H_2} x 10³ (corr.)	Metal Analysis, Wt Pct S	Al	log K'	log f_S	log f_S°	log f_S^{Al}
A-1	1.03	0.351	1.93	2.535	0.044	−0.010	0.054
A-2	1.00	0.302	1.91	2.480	0.097	−0.008	0.105
A-3	0.95	0.256	2.78	2.431	0.146	−0.007	0.153

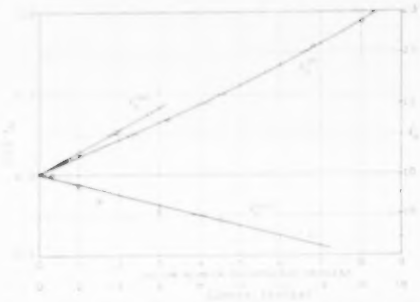

Fig. 3—Effects of aluminum, phosphorus, and copper on activity coefficient of sulphur.

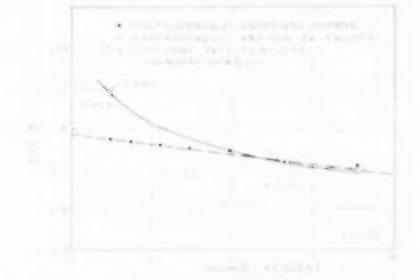

Fig. 4—Equilibrium in Fe-S and Fe-S-Cu alloys.

Fe-S-P—Similar data on melts containing phosphorus have been published recently by the authors[5] and are repeated here for comparison with other data. They are shown in Table II and Fig. 3. Again there is no apparent dependence on sulphur content and $\log f_s^P$ is linear up to about 5 pct P.

Fe-S-Al—Three melts were made containing aluminum and the resultant data are shown in Table III and Fig. 3. Again $\log f_s^{Al}$ is linear.

Fe-S-Cu—The data of Rosenqvist and Cox both on pure iron and on Fe-Cu alloys are inconsistent with the foregoing data at low sulphur concentrations, but the agreement is good above 2 pct S. This is shown in Fig. 4 where their data on Fe-S alloys at 1550° are compared with those of Sherman, Elvander, and Chipman at 1540°. Their points for copper-bearing alloys are also shown, with the percentage of copper indicated. The value of $\log f_s^{Cu}$ is taken as the vertical distance of each of these points below the line representing their values for the binary solutions. These are plotted against concentrations of copper in Fig. 3. The line is drawn straight since the precision is inadequate to establish the shape of the curve.

Fe-S-Si—As a further basis of comparison with

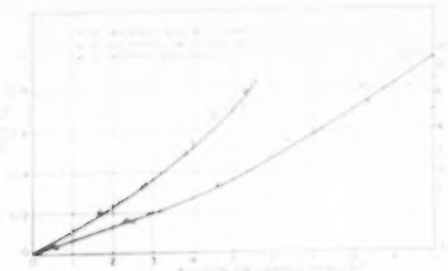

Fig. 5—Effects of silicon and carbon on activity coefficient of sulphur

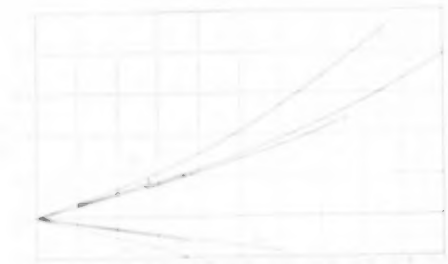

Fig. 6—Comparison of the effects of several elements on the activity coefficient of sulphur.

the work of Morris and Williams, a number of heats were made in our apparatus by David B. Smith using Fe-S-Si alloys. The heats were made in silica crucibles to avoid any errors due to reduction of the crucibles. The results are shown in Table IV and Fig. 5. Originally aimed at 1615 C, it was found subsequently that the actual temperature had been 1630 C. It is unlikely that this difference has an appreciable effect on f_s,[10] provided only that the value of K appropriate to the temperature is used in the computation. The results agree remarkably well with the data of Morris and Williams and the curve is drawn to represent an average of the two sets of data.

Fe-S-C.[11] The data of Morris and Buehl for Fe-S-C alloys containing small amounts of aluminum may be corrected to correspond to the simple ternary in the following manner. The original values of f_s are divided by f_s^{Al} read from Fig. 3 to obtain the corrected values. This is an empirical procedure whose justification will be discussed under quaternary systems. The resultant values of log f_s^C are shown in Fig. 5.

Comparison. In the foregoing calculations the percentage by weight has been used rather than the atomic concentration because of its ready application in steelmaking. For a comparison of the effects of the several elements, it is more enlightening to use atom fractions. In this calculation, let the activity coefficient be represented by $\gamma_s = a_s/N_s$ and let γ_s approach unity as N_s approaches 0. Values of γ_s differ only slightly from those found for f_s and analogous equations are applicable; for example, $\gamma_s = \gamma_s' \cdot \gamma_s^{Si}$. Calculations were made in the same manner as before, after changing all percentages to atom fractions. The results are shown in Fig. 6. The effects of phosphorus, carbon, aluminum and silicon are similar, increasing in the order named. Manganese has a greater effect than copper.

At small concentrations each of the lines is straight and the slope in this region is of possible theoretical interest. The slopes are shown in Table V, together with the approximate limit below which each line is straight. Values are given for the coefficient $\partial \log f_s/\partial$ [% third component] read from Fig. 1, 2, 3, and 5 and for the quantity $\epsilon = \partial \ln \gamma_s/\partial N_x$.

Quaternary and More Complex Systems

The simultaneous effects of two or more alloying elements on the activity coefficient of sulphur have been studied in a series of experiments at 1600 C whose results are recorded in Table VI. These experiments were designed to establish a method which could be used for computing activity coef-

Table IV Equilibrium Data on Fe-S-Si Alloys at 1630 C
Experimental work by David B. Smith

Run No.	P_{H_2O}/P_{H_2} x 10³ (corr.)	Metal Analysis, Wt Pct S	Si	log f_s	log γ_s	log f_s	log γ_s
2B1	1.60	0.102	0.34	2.513	0.340	0.005	0.511
3C4	2.91	0.187	0.50	2.631	0.320	0.006	0.734
3C5	2.91	0.170	0.92	2.390	0.273	0.005	0.778
3C6	2.91	0.160	2.31	2.205	0.024	0.005	0.928
3CM	2.91	0.197	2.34	2.259	0.000	0.005	0.905
4A6	2.45	0.046	0.52	2.421	0.330	0.010	0.156

Table V Effects of Small Concentrations of Added Substances On Activity Coefficient of Sulphur

Third Component	$\partial \log f_s$ / ∂ [% Third Component]	Limit [% Third Component]	$\epsilon = \partial \ln \gamma_s$ / ∂N_x
C	+0.111	1.1	+5.6
Si	+0.065	3.5	+7.5
P	+0.043	4.0	+5.5
Al	+0.054	3+	+6.0
Cu	+0.013	13+	3.4
Mn	0.025	5.0	5.7

Table VI Equilibrium Data on Complex Alloys at 1600 C

Run No.	P_{H_2O}/P_{H_2} x 10³ (corr.)	Metal Analysis, Pct S	C	Al	P	Si	Mn	log f_s Obs.	log f_s Calc.
CA-1	1.19	0.309	0.56	2.16				0.176	0.17
2	1.42	0.311	1.04	2.21				0.217	0.23
3	1.74	0.289	1.93	2.08				0.357	0.35
4	1.43	0.397	0.74	1.14				0.133	0.14
5	1.68	0.251	1.40	1.09				0.210	0.21
6	1.63	0.244	2.72	1.10				0.402	0.39
F-9	0.938	0.242	0.95	0.19	1.10			0.166	0.16
10	1.19	0.262	2.01	0.25	1.06			0.346	0.33
11	0.978	0.196	1.96	0.27	0.97			0.296	0.29
12	0.778	0.177	0.99	0.23	2.03			0.218	0.21
B-1	1.08	0.277	1.01	0.17		0.09		0.168	0.18
2	1.10	0.215	1.91	0.19		0.86		0.286	0.29
3	0.923	0.175	0.93	0.20		2.09		0.249	0.26
4	1.51	0.190	2.10	0.12		1.95		0.398	0.41
5	2.23	0.254	2.17	0.28		2.87		0.520	0.54
6	2.70	0.233	2.08	0.26		4.05		0.641	0.67
B-7	0.647	0.222	0.99	0.09			1.14	0.041	0.03
8	0.992	0.313	0.74	0.12			2.87	0.016	0.01
9	1.07	0.429	0.46	0.10			3.21	0.027	0.03
10	0.743	0.187	2.03	0.18			3.04	0.176	0.16
11	1.30	0.266	2.64	0.17			2.59	0.266	0.25
12	1.19	0.345	1.55	0.14			2.76	0.115	0.10
B-13	0.825	0.311	0.38	0.15		0.60	3.50	0.000	0.00
14	0.808	0.298	0.50	0.07		0.48	3.26	0.008	0.00
15	0.806	0.400	0.17	0.11		0.40	2.72	0.027	0.02
16	0.730	0.281	0.30	0.08		0.49	3.01	0.008	0.01
17	0.823	0.253	0.98	0.04		0.75	2.88	0.090	0.08
18	0.924	0.190	2.01	0.12		1.00	2.56	0.204	0.25
B-19	1.68	0.200	1.03	0.31	1.52	3.95	2.17	0.301	0.32
20	1.66	0.244	1.26	0.27	1.93	2.16	3.80	0.459	0.43
21	1.50	0.198	1.55	0.19	2.77	1.33	2.99	0.395	0.38

ficients throughout the range of compositions encountered in iron and steel practice. The method of computation is explained below and the calculated value of log f_s is shown for comparison in the final column of the table.

A simple method for combining activity coefficients, applicable to dilute solutions, has been applied[7] to an analogous case of oxygen in steel. It rests upon a simple extension of eq 6 to multicomponent systems:

$$f_s = f_s' \cdot f_s'' \cdot f_s''' \cdot \ldots \cdot f_s^{(n)} \quad [7]$$

or more conveniently,

$$\log f_s = \log f_s' + \log f_s'' + \log f_s''' + \ldots + \log f_s^{(n)} \quad [8]$$

While this equation is thermodynamically valid for an extremely dilute solution of components 2, 3, 4, n, in component 1, its application to solutions of higher concentration is semi-empirical and its usefulness can be estimated only from experiment.

The experimental data on log f_s in binary and ternary systems are assembled in Fig. 7, which will serve as a working graph. The simple addition of values of log f is illustrated for three experiments in the following:

	CA-1	CA-4	CA-6
Log f_s'	−0.509	−0.009	−0.006
Log f_s''	−0.061	−0.221	−0.320
Log f_s'''	−0.120	−0.115	−0.059
Log f_s''''	−0.172	−0.327	−0.373
Log f_s (obs.)	−0.176	−0.357	−0.402

The agreement between observed and calculated values is very good in the first example but somewhat outside the normal limits of experimental error in the other two. A more accurate method for combining the data is desirable.

The basis for a better method is found in an observation of Morris and Buehl: "One experiment of an exploratory nature was run at 1600 C to determine the effect of carbon and silicon when present together in the melt. In this test the addition of 1.40 pct silicon to a melt containing 4.0 pct carbon increased the activity coefficient of sulphur from 3.75 to 5.08. This increase is about the same as would have resulted from adding 1.4 pct silicon to an iron-silicon melt in which γ_s was initially 3.75, that is, adding 1.4 pct silicon to iron containing 7.15 pct silicon."

An empirical method utilizing this principle which serves sufficiently well for obtaining f_s in a complex alloy depends upon the order in which individual values of log $f_s^{(n)}$ are added. Beginning with the straightest lines of Fig. 7 and proceeding to those of successively greater curvature, the method is as follows. The value of log $f_s'^{(n)}$ is read. A point on the phosphorus curve is located having equal value of log f_s. To the phosphorus content of this is added that of the alloy, and the corresponding value of log $f_s'^{(n)}$ is read. A point on the carbon curve is located having log f_s equal to this last value. To the carbon content of this is added that of the alloy, and the corresponding value of log $f_s'^{(n)}$ is read. This procedure is repeated for the silicon line, adding the silicon content of the alloy. This yields a value of log f_s which represents the sum of all the positive contributions. The negative contributions are added similarly in the order sulphur, copper, and manganese and are subtracted from the previous sum.

Table VII. Activity Coefficient of Sulphur in Pig Iron

No.	Si	P	Mo	C	f_s
1	0.5	0.2	0.5	4.8	5.5
2	1.0	0.5	1.0	4.6	5.6
3	2.0	0.5	1.0	4.3	6.3
4	4.0	0.5	1.0	3.7	8.5
5	1.0	2.0	1.0	3.8	6.0
6	1.0	0.2	3.0	4.7	5.4

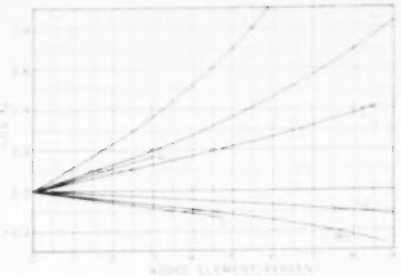

Fig. 7—Graph for computing log f_s in complex systems.

The calculation is illustrated for the three examples used above as follows:

	CA-1	CA-4	CA-6
Log $f_s'^{(n)}$	−0.120	−0.115	−0.059
Equivalent pct C	1.03	1.03	0.60
Equivalent total C	1.84	2.90	3.32
Log $f_s'^{(n)}$	−0.124	−0.357	−0.396
Log f_s'	0.009	0.009	0.006
Log f_s (calc.)	−0.175	−0.348	−0.390
Log f_s (obs.)	−0.176	−0.357	−0.402

The discrepancy between the calculated and the observed value has been reduced to about one third and is now within the limits of experimental error. This is the method that has been used in obtaining all the calculated values of log f_s in Table VI. In dilute solutions where the curved portions of the lines are not reached, it is equivalent to simple addition of values of log f_s. At higher concentrations the results depend upon the order of addition especially of carbon and silicon, and the order chosen is based principally on trial and error.

Sulphur in Pig Iron

The activity coefficient of sulphur in pig iron can be calculated by the method used above, but the data do not go to high enough percentages to include spiegel or silvery iron. For several pig iron compositions, the results are shown in Table VII. The carbon concentration of pig iron is usually lower, the higher the silicon and phosphorus content. For purposes of calculation, hypothetical carbon contents have been used which correspond approximately to graphite saturation at 2700 F. The results show an activity coefficient of 5.4 to 6.3 for ordinary grades, with higher figures at higher silicon content. The significance is that in pig iron sulphur should be much more easily removed than in mild steel because it has about 5.5 times the escaping tendency.

Summary

The chemical behavior of sulphur in liquid iron at 1600 C is investigated through study of the equilibrium:

$$H_2 + \underline{S} = H_2S; K = (p_{H_2S}/p_{H_2} \cdot a_s)$$

From the known equilibrium constant of the reaction and the experimental data, the activity of sulphur in the melt is determined.

The activity coefficient of sulphur defined as $f_s = a_s/N_s$, S is increased by phosphorus, carbon, aluminum, and silicon, named in order of increasing effect per atom. It is decreased by copper and manganese, the latter having the larger effect.

A semi-empirical method is devised for calculating the activity coefficient of sulphur in complex solutions of four or more components. This method is tested against extensive data on solutions of this kind.

The activity coefficient of sulphur in iron of typical blast-furnace composition is about 5.4 to 6.3 and increases with increasing silicon.

Acknowledgments

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Technical Note

Diffusion and Precipitation of Carbon in Some Alloys of Iron

by Charles Wert

THE diffusion and precipitation of carbon and nitrogen in α iron have previously been investigated using the internal friction as a measuring tool.[1] Most of this work has been done on rather pure iron; no effect was observed which would indicate that either of these phenomena is affected by the rather small amounts of impurity found in these samples of iron. Fast[2] observed, however, that both the diffusion rate and the aging properties of nitrogen in iron were sensibly changed by the addition of ½ pct Mn. The diffusion rate at a given temperature is reduced and the nucleation of nitrides is apparently changed somewhat. He observed no such changes for carbon in this alloy. Later Dijkstra and Slade[3] extended these observations on nitrogen to other alloys of iron. They found that metals other than manganese also influence the behavior of nitrogen in iron; chromium, molybdenum and especially vanadium affect the diffusion and precipitation of nitrogen even more than manganese does. Their results have been verified in all essential details by this author, who finds in addition that small additions of nickel have about the same effect on the diffusion of nitrogen as the same amount of manganese.

The present work was undertaken to study the effect on carbon in iron of these alloying elements. The alloys used had two sources. The material used as a "standard" (designated Fe in this paper) in the measurements was some iron supplied by J. K. Stanley, of Westinghouse Electric Corp., who reported 0.01 pct O as the only major impurity. He also made up an alloy of 0.26 pct Ni and one of 0.9 pct Mo using this same iron as a base. They were supplied by him in 2 lb billets, which were sawed into rods and cold formed into 0.030 in. wires for measurement. The alloys of chromium, manganese, and vanadium were prepared by the metallurgy section of the Institute for the Study of Metals, University of Chicago, by adding these metals in turn to vacuum melts of National Research Corp. iron. The small castings prepared in this way were also cold formed into 0.030 in. wires. After these wires had been prepared they were decarburized for 30 min at 730°C in wet H. After this treatment, no trace of carbon (as measured by internal friction) could be found. All specimens were then carburized for about 15 min at 710°C and water quenched. By this treatment the internal friction at 40°C was raised to a $Q^{-1} \sim 0.015$.

The diffusion of carbon in these alloys was studied by observing the shape and position of the internal friction peak at 0.9 cycles per sec. These measurements were made using a torsional pendulum in a manner described earlier.[1] The data are plotted in Fig. 1. It is seen that there is no apparent difference in internal friction between the various alloys. This is in marked distinction to the broadening and shift-

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Table I. Internal Friction in Various Steels

Alloy	Carbon Content by Chemical Analysis, Pct	Internal Friction
Fe	0.0143	0.015
Fe + 0.5 pct Ni	0.0305	0.015
Fe + 0.9 pct Mo	0.027	0.0155
Fe + 0.5 pct Cr	0.0165	0.015
Fe + 0.5 pct Mn	0.065	0.014
Fe + 0.5 pct V	0.038	0.0155

ing of the nitrogen peak in these alloys.[2,3] It would appear from Fig. 1 that these alloying elements in these concentrations have only a small effect on the diffusion of carbon. There is a chance, of course, that there are carbon atoms which are trapped in interstitial sites adjacent to impurity atoms. These carbon atoms might not contribute to the damping in the neighborhood of the normal carbon peak. However, if this is the case, then there should be a relaxation peak at some other temperature for these carbon atoms (as was observed for the "trapped" nitrogen atoms in these alloys[3]). No such peak was found for carbon in any of these alloys between the temperatures −35° and 200° C. It must be concluded from these measurements, therefore, that the diffusion rate of carbon at 40° C in α iron is the same as that in these alloys.

The precipitation measurements were carried out at 150° C. The course of the precipitation was followed by observing the decrease in the internal friction peak at 40° C. This method has been adequately described previously.[1] The data are shown in Fig. 2. All the alloys with the exception of the vanadium alloy follow closely the same pattern. It must be the case, therefore, that the alloying elements manganese, chromium, molybdenum, and nickel in these concentrations do not affect the precipitation of carbon at this temperature. For the alloy of 0.5 pct V in iron, a considerable effect is observed. Here the precipitation rate is roughly ten times faster than for the others. Since the diffusion rate of carbon in Fe + 0.5 pct V does not differ from that for the other alloys (as my interpretation of Fig. 1 indicates), the nucleation of some carbide must be aided by the presence of vanadium. The carbide which is formed should be more finely dispersed than in the other materials. This would allow precipitation to go on faster, as the data in Fig. 2 require.

The internal friction method of carbon analysis will only indicate how much carbon is in solid solution. It will not tell how much carbon might be tied up in the various carbides. To get a complete picture of the alloys, then, it is necessary to have an analysis for total carbon content made by some kind of chemical analysis. For the alloys reported here, this has been done; the results are presented in Table I.*

* For this analysis, the author is indebted to Dr. George Wetmer of the magnetics department, Westinghouse Research Laboratory.

The first thing to be observed in Table I is that for the purest iron used in this work, the agreement between internal friction and actual carbon content is very good. This is the first direct proof that the internal friction technique is a valid method for carbon analysis in iron. For the other alloys the agreement is bad. At the moment, no reasonable explanation can be made for this disagreement. If the excess carbon is in solid solution but is trapped near the alloying elements, the internal friction

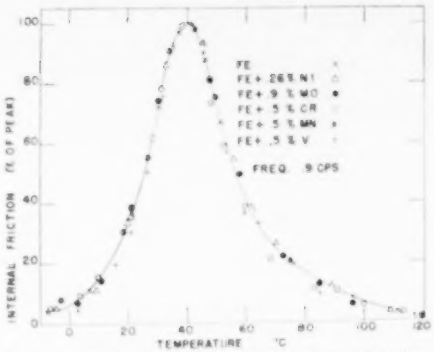

Fig. 1—Internal friction peak for C in Fe and some of its alloys.

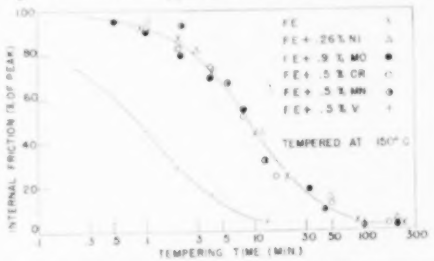

Fig. 2—Precipitation of C from supersaturated solid solution in iron and some of its alloys.

peak of Fig. 1 should be modified as for N in these alloys.[2,3] If the carbon is bound up in carbides which are formed at the carburizing temperature of 710° C, then nickel (which has no stable carbide at this temperature) should not exhibit such a disagreement as the strong carbide formers.

Even with these disturbing factors, a number of worthwhile conclusions may be drawn from the data: 1—For pure iron the internal friction is a reliable indicator of carbon content; this adds credence to the earlier use of it in study of aging of carbon in iron. 2—The presence of Cr, Ni, Mn, V, and Mo in amounts of about ½ wt pct does not affect the relaxation peak of carbon in α iron. These elements in this amount presumably do not sensibly alter the diffusion rate of carbon in iron. 3—With the exception of vanadium, these alloying elements do not affect the rate of precipitation of carbon in α iron. Vanadium hastens the precipitation of carbon; this is tentatively interpreted as indicating that it provides more precipitation nuclei for the carbide. These last two conclusions must be qualified by the remark that there is in some of these alloys some carbon tied up in some unknown way. The presence of this carbon seems in no way (with the exception of the vanadium alloy) to affect the behavior of the "free" carbon.

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Solid Solubility of Sulphur in Iron

by Terkel Rosenqvist and Boleslaw L. Dunicz

IT has long been suspected that sulphur has a small but finite solid solubility in iron, but up to the present more accurate data have been lacking. The survey given by Hansen[1] illustrates the divergence of data and opinions. Solubilities between 0.01 and 1.5 pct have been suggested, and opinions vary regarding the effects of temperature and phase transformations in iron on the sulphur solubility.

The extent of solid solution and its variation with temperature might be expected to be an important factor in the occurrence of hot shortness in steel. Therefore, the object of the present investigation was to study this problem more systematically. It was furthermore hoped that solubility data for sulphur might contribute to an understanding of the factors which determine solid solubilities in general.

Experimental Procedure

The present work was based on the measurement of the chemical activity of sulphur in the solid solution by means of equilibrium with a mixture of hydrogen and hydrogen sulphide. When the solid solubility limit is reached, the sulphur activity in the iron becomes equal to that in the coexisting sulphide phase.

A gas mixture of a controlled H_2S/H_2 ratio was passed over pure metallic iron heated to a temperature in the range 900° to 1500°. The amount of sulphur absorbed by the iron was determined by analysis, and from a plot of percentage of sulphur vs. gas composition the sulphur activity and the solubility limit were determined.

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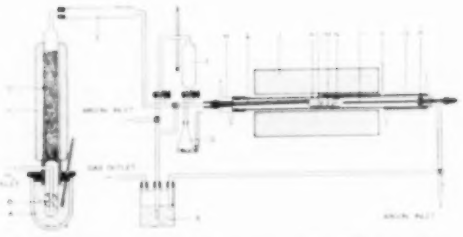

Fig. 1—Apparatus for H$_2$-H$_2$S generation and equilibrium studies.

A	— Silicone oil thermostat	K	— Al_2O_3 thimble
B	— Sulphur bath	L	— Al_2O_3 boat
C	— Furnace, 500° C	M	— Iron samples
D	— Alundum chips	N	— Al_2O_3 baffle
E	— P_2O_5	O	— Alundum rod
F	— Gas sampler, 118.5 cc	P	— Thermocouple
G	— Gas absorption flask	Q	— Watercooling
H	— Watercooling	R	— Asbestos plug
I	— Al_2O_3 capillary	S	— Phosphoric acid
J	— Reaction furnace		

As the rate of diffusion of sulphur in solid iron is very small, often several days were needed to be sure that equilibrium conditions had been established. To produce a gas flow with less than 1 pct H_2S of constant composition for several days, was in itself a problem. The most satisfactory arrangement was obtained by a continuous synthesis of hydrogen sulphide from the elements. This was done in the part of the apparatus shown to the left in Fig. 1.

The hydrogen used was first purified in tank batches by passage through activated charcoal cooled in liquid nitrogen under a pressure of 150

atm. This was done in order to remove any hydrocarbons which later might have contaminated the iron with carbon. The gas was further purified by passage through heated Pd-asbestos and was dried over $CaCl_2$ and P_2O_5.

The purified hydrogen was bubbled through molten sulphur kept at some temperature between 140° and 180°C (*A*, Fig. 1) in a silicone oil thermostat. The hydrogen became saturated with sulphur vapor but did not react chemically with it. This gas mixture was then passed directly into a reaction tower filled with Alundum chips and kept at a temperature of about 500°C. Here the sulphur vapor reacted with hydrogen to give hydrogen sulphide. By varying the temperature of the constant temperature bath, the H_2S/H_2 ratio could be varied between 10^{-1} and 10^{-5}. For a fixed geometry of the bath, its heater and temperature controller, the gas composition was kept constant to within 2 or 3 pct for several days.

The mixture of hydrogen and hydrogen sulphide was dried over P_2O_5 and was passed through a gas sampler of known volume (*F*, Fig. 1), before it entered the main reaction furnace. For gas analysis this volume was closed off, and the trapped gas was flushed by means of argon into an absorption flask containing alkaline potassium hypochlorite solution. The amount of hypochlorite needed to oxidize the hydrogen sulphide to sulphate was determined by iodometric titration, in the way recently described by the present authors.[5] From the known volume, temperature, and pressure of the gas sample, the percentage of H_2S could be calculated and the H_2S/H_2 ratio determined.

In Fig. 2 are plotted all gas ratios obtained over a period of several months as a function of the temperature of the molten sulphur. The dashed curve represents the gas composition calculated from the vapor pressure of liquid sulphur as given by West and Menzies.[6] This curve is calculated under the assumption that the vapor consisted of S_8 molecules and that the hydrogen pressure was always 750 mm Hg. The slightly lower values for the experimental points may be due to incomplete vapor saturation, but also may be because the vapor, in addition to S_8 molecules, contained some S_2 molecules. The scatter may be due partly to variations in atmospheric pressure, which affected the H_2 pressure.

From the gas sampling tube the gas passed into the reaction furnace at a flow rate of 50 to 100 cc per min. The reaction furnace had a molybdenum resistance winding, protected against oxidation by a hydrogen-nitrogen atmosphere. The temperature was controlled by a heavy Pt-Pt-Rh thermocouple connected with a Leeds and Northrup "Micromax" temperature controller. The temperature setting was calibrated by means of a standardized thermocouple which, between runs, was inserted into the furnace to the point where the iron samples usually were heated. The temperatures are regarded as accurate to 2° at 900° to 1000°C and 5° at 1500°C.

In order to prevent thermal diffusion in the gas mixture, several precautions were taken. First the gas was mixed with about an equal amount of argon, purified by passage over KOH, P_2O_5, and heated copper gauze. This is in accordance with the finding by Dastur and Chipman[4] that an inert heavy gas prevents thermal diffusion of the lighter gases. Second, the inlet to and the outlet from the hot zone were constricted. The boat containing the iron

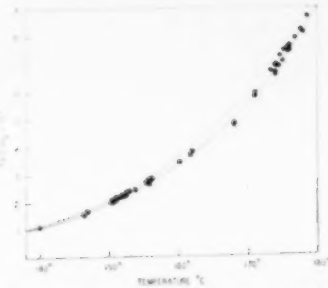

Fig. 2—H_2S/H_2 ratio vs. the temperature of molten sulphur. The dashed curve is calculated from the vapor pressure assuming S_8 molecules and P_{H_2} = 750 mm Hg.

samples was placed in the hot zone inside a chamber consisting of a thimble with a capillary inlet in the bottom. The gas outlet was constricted by means of a smaller tight fitting thimble which also acted as a heat shield.

In order to prevent contamination of the iron with silicon, all refractories were of pure sintered aluminum oxide. This included the main reaction tube as well as the thimbles, the capillary tube, and the boat for the samples. The boat was attached by means of a molybdenum wire to an Alundum rod which projected from the furnace, and which made it possible to pull the boat out into the water-cooled zone.

The boat was loaded usually with three iron samples. These were made from Puron iron, cold-rolled to a thickness of 0.003 in. Strips of this foil were coiled up into spirals and were placed in the boat so that the gas could flow through the spirals and make intimate contact with the entire surface area. Each iron sample weighed 1 to 1.5 g.

After the iron samples had been heated in the gas flow for a certain time, the boat was pulled out into the cold zone in a counterflow of cold, purified argon which replaced the H-H_2S atmosphere. No attempt was made to quench the iron samples, since only the sulphur concentration and not the structural distribution of sulphur in the cooled samples was of interest in this work.

The sample in the middle of the boat, No. 2, Fig. 1, was usually taken out for analysis. The run was continued with sample No. 1 moved to the position of No. 2, and No. 3 to the position of No. 1; a fresh iron sample was put in the vacant place. In this

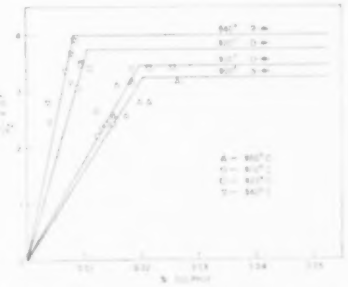

Fig. 3—Sulphur content vs. H_2S/H_2 ratio, 900° to 940°C.

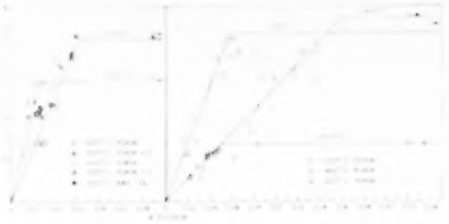

Fig. 4—Sulphur content vs. H.S/H. ratio, 1000° to 1500° C.

way it was possible to have the samples heated in the gas flow for various lengths of time.

In some cases, iron samples with about 0.15 pct S were used in order to approach the equilibrium from the side of high sulphur content. These iron samples were prepared from the Puron spirals which were heated in an evacuated and sealed quartz tube with a calculated amount of sulphur at 900° C for several days. A plot of the sulphur content of the samples vs. reaction time, for 1200° C, showed no systematic variation with time between 20 and 110 hr, and the samples with high initial sulphur content gave the same result as the initially pure iron. At 910° C, 48 hr were sufficient to give equilibrium values. At 1350° to 1500° C, the runs lasted for 12 to 36 hr.

Some runs at 1200° C were made with a special high purity iron which had been prepared by the National Bureau of Standards.* This iron was

* Thanks are due to J. G. Thompson for this material.

claimed to be more than 99.99 pct pure. As will be shown in Fig. 3, this material gives the same results as Puron.

The sulphur content of the iron samples was determined by dissolving the samples in 6 N hydrochloric acid and absorbing the evolved hydrogen sulphide in potassium hypochlorite solution, which again was titrated in the same way as for the gas samples.

The impurities in a number of samples were determined by semiquantitative spectrographic analysis. Some typical analyses are given in Table I. It should be noted that these data are regarded as accurate only to within a factor of three.

It may be noted that for samples treated up to 1400° C the total amount of metallic impurities was between 0.01 and 0.1 pct, whereas for the sample treated at 1500° C the silicon content was of the order of 1 pct.

The oxygen content was determined in a few samples treated at 940° C in the regular way, with the exception that the H_2S concentration in the gas was very low ($< 10^{-3}$). The oxygen content was determined by the vacuum fusion method and the following values were obtained:

Puron heated for 48 hr: 0.034 pct O.
N.B.S. heated for 24 hr: 0.024 pct O.
N.B.S. heated for 48 hr: 0.018 pct O.

The carbon content of the samples was not determined, but since the gas was virtually free of hydrocarbons, it is believed to be very low.

It is difficult to predict to what extent these impurities will effect the sulphur solubility; though it is probable that some effect will be present.

Experimental Results

In Figs. 3 and 4 curves are plotted for the sulphur content of the iron vs. the H_2S/H_2 ratio of the gas for a number of temperatures between 900° and 1500° C. The abscissas are drawn to different scales for the different temperatures. For all temperatures the saturation point is characterized by a sudden increase in the sulphur content at a certain constant gas composition. When the gas ratio was increased beyond this point, the iron was gradually transformed into FeS or a sulphide-rich melt.

Below the saturation point the experimental data show considerable scatter. This scatter is larger than the probable error in the gas and metal analysis, and its reason cannot be easily given. It cannot be due to incompletely established equilibrium, since the sulphur content does not vary systematically with the duration of the run. It may be due to varying amounts of impurities which may affect the activity coefficient of dissolved sulphur even when they are present in very small amounts. Another possibility is that some of the sulphur is not present in true solid solution, but is absorbed in the grain boundaries. In such a case the sulphur content might be a function of the size and shape of the grains. For the lack of any good explanation, averages of the experimental points are taken to determine the solubility limits. These are listed in Table II.

At 1350° C some points, connected by a broken line, seem to reach a horizontal level at a lower gas ratio than the rest of the points. It will be shown later that sulphur depresses the A_3 point (the γ-δ transformation) to about 1365° C, and it is conceivable that small amounts of impurities, e.g., silicon or molybdenum, in some samples might have

Table I. Material

Element	As Received		N.B.S. 1200° C	Puron			
	N.B.S.	Puron		950° C	1000° C	1200° C	1500° C
Si	0.004	0.005	0.002	0.001	0.001	0.01	0.8
Cu	0.005	0.005	0.002	0.0002	0.001	0.01	0.002
Ni	0.001		0.001				
Mo	0.002	Trace	0.001	0.001	Trace	Trace	0.01
Mn	0.005			0.02			0.02
Al	0.001	Trace		Trace	Trace	Trace	Trace
Mg	0.001	Trace		Trace		Mo—0.001	Cr—0.005

lowered the transformation temperature to below 1350°C. In that case the broken, horizontal line should correspond to the two-phase region $\gamma+\delta$ Fe. Since these samples were consumed by the sulphur analysis, their purity has not been determined.

In Fig. 5 the variation of the gas ratio for constant sulphur contents is given as a log H_2S/H_2 vs. $1/T$ plot. The upper boundary of the solid solubility region corresponds to the presence of the two phases, iron and solid or molten sulphide. This curve is in agreement with independent, and as yet unpublished, equilibrium measurements on samples with 10 to 20 pct S measured up to 1350°C. In the upper right corner of Fig. 5, the equilibrium between solid iron sulphide and sulphide melt is indicated.

To the left in Fig. 5 are drawn, for comparison, the H_2S/H_2 ratios for low sulphur melts as determined by Sherman, Elvander, and Chipman.[7] The boundary between these melts and the δ region is constructed from their data combined with existing literature data for the liquidus curve.[8]

It is evident from Figs. 3 and 4 that the solubilities at 910°C (α Fe) and 1400°C (δ Fe) are distinctly higher than in γ Fe at 920° and 1350°C, respectively. This indicates that sulphur raises the A_3 point and depresses the A_4 point. Because of the insufficient precision of the gas equilibrium method, it was regarded as impractical to search for the change in the transformation point by further experimental studies. Instead the changes in transformation temperatures were calculated from the compositions of the coexisting phases and the heats of transformation. The change in the transformation temperature, ΔT, is given by the equation:

$$\Delta T = -\frac{RT^2}{\Delta H_{tr}} \cdot \frac{\Delta[S]}{100}$$

Here ΔH_{tr} denotes the heat of transformation, which according to Olander[9] is 220 cal for the A_3 point and 280 cal for the A_4 point. $\Delta[S]$ is the difference in sulphur content of the two coexisting phases in atomic percent. This equation is valid for dilute solutions and relatively small values for ΔT.

From the curves in Figs. 3, 4, and 5, the compositions of the coexisting phases at sulphur saturation can be determined. For the A_3 point, around 910°C, these compositions are 0.020 wt pct for α Fe and 0.007 wt pct for γ Fe, which gives $\Delta[S]$ equal 0.023 atomic pct, corresponding to an increase of the A_3 point by 3°, i.e., to about 913°C. For the A_4 point, between 1350° and 1400°C, the maximum solubilities are 0.16 to 0.18 wt pct for δ Fe and 0.06 wt pct for γ Fe. This makes $\Delta[S]$ equal to about 0.20 atomic pct, corresponding to a lowering of the A_4 point by 35° to 40°, i.e., to about 1365°C.

Table II. Saturation Limits

Temperature, °C	Wt Pct S	H_2S/H_2, 10⁻³
900	0.020±0.002	3.25±0.10
910	0.020±0.002	3.45±0.10
920	0.011±0.002	3.75±0.10
940	0.008±0.001	4.90±0.10
1000	0.013±0.003	5.60±0.10
1200	0.031±0.005	7.30±0.10
1350	0.058±0.01	7.80±0.2
1400	0.16 ±0.01	8.45±0.2
1500	0.05 ±0.01	2.65±0.2

The boundaries between the α and γ, and between the γ and δ phase regions are drawn in Fig. 5 in accordance with these calculations.

It will be noted that the curves for equal sulphur concentrations are drawn continuously through the α–γ boundary, whereas theoretically the change should be discontinuous. This is because the curves are drawn through the experimental points. It is likely that small amounts of impurities have widened the transition range and made it appear to be less discontinuous than in the pure binary system. The calculated value for the rise in the A_3 point, however, applies for the pure system.

It is now possible to calculate standard free energies for the solution of sulphur in iron referred to hydrogen and hydrogen sulphide. For this purpose the standard state of dissolved sulphur is chosen to be a hypothetical, ideal, 1 pct solution. The standard free energy, ΔF°, for the reaction $H_2S = H_2 + S$(dissolved) is then given by the equation:[10]

$$\Delta F^\circ = -RT\ln\frac{H_2 \cdot \text{pct S}}{H_2S}$$

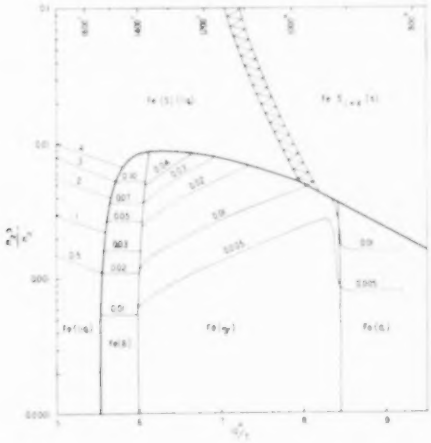

Fig. 5—Log H_2S/H_2 vs. $1/T$ for constant compositions.

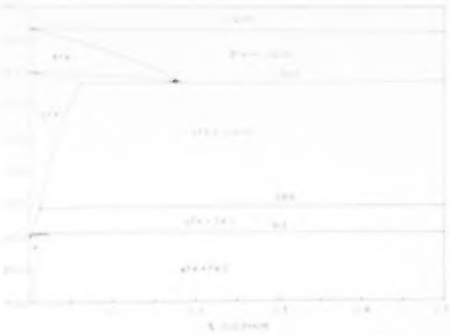

Fig. 6 — Phase diagram for the low sulphur region of the system iron sulphur

In order to obtain the free energy as a linear function of the temperature, it is assumed that the heat of reaction is independent of temperature, i.e., that the curves for equal concentration in Fig. 5 are straight lines. Actually the curves for sulphur in γ Fe, which were drawn through the experimental points, are slightly curved. But the experimental data hardly justify this curvature, and, therefore, for the calculation of the free energy, an average slope is chosen.

Then for the solution of sulphur in γ Fe, ΔF equals 14 700 — 12.95 T, for sulphur in δ Fe, ΔF equals 0 — 5.76 T cal.

Whereas the values for the free energies are regarded as correct to within 500 cal, the first term, the heat of solution, is accurate only to within 2000 cal, and the second term, the entropy, only to within one entropy unit. The absence of any heat of solution for sulphur in δ Fe applies only within these limits.

For comparison the free energy of sulphur in liquid iron is given according to Sherman, Elvander, and Chipman:[5]

$$\Delta F = 9840 - 6.54\ T \text{ cal}$$

It may be noticed that whereas the entropy term is rather constant, the heat of solution varies greatly between the three phases. This corresponds to the structural picture that the randomness of distribution of sulphur in iron is about the same in the three cases, whereas the binding energy differs greatly.

Phase Diagram

It is now possible to draw a phase diagram for the low sulphur region of the system Fe-S, Fig. 6. The shape corresponds in principle to the schematic diagram suggested by Wever and to the qualitative observation by Heindl that sulphur depresses the temperature of the α-δ transformation. One of the remarkable features of this diagram is that the solid solubility in γ Fe increases with increasing temperature even above the eutectic temperature. This is undoubtedly a result of the strong positive deviation from ideality in the melt. The melt is almost at the point of breaking down into two immiscible liquids, and the abnormal shape of the liquidus curve is reflected in the solidus curve.

This system is one of the very few which have a contracted γ region. The nonvariant horizontal at 1365°C has no accepted name in metallurgical literature, although the term eutectic has been used colloquially.

Taking for example, iron with about 0.1 pct S, it is perfectly solid at 1400°C. By cooling below 1365°C, γ Fe is formed and some liquid phases extruded. By further cooling, more liquid sweats out until the eutectic melt solidifies at about 988°C. It is conceivable that this will have considerable bearing on the hot shortness of steel as far as the liquid phase might be finely divided along the grain boundaries.

It should be pointed out here that the solubility data which have been obtained for pure iron may not be applied directly to commercial steel which contains various amounts of C, Si, Mn, Cu, Ni, etc. It may be estimated in which direction these impurities will affect the solid solubility. Carbon and silicon will preferentially be dissolved in the solid iron phases and may here affect the activity coefficient of dissolved sulphur. Analogous to what has been found for molten iron[5, 6] it might be expected that the activity coefficient would increase, i.e., the slope of the slanting part of the curves in Figs. 3 and 4 would be increased.

Manganese, copper, and nickel will be preferentially dissolved in the sulphide phase, which thereby will be stabilized. This means that the level of the horizontal part of the curves in Figs. 3 and 4 will be lowered. In all cases, the alloying elements will tend to decrease the sulphur solubility. Most probably the general appearance of the phase diagram will be the same, and the conclusions reached as to the effect of sulphur on the phase transformations, and the variation of the solubility with temperature, will still apply.

Acknowledgment

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Titanium-Aluminum System

by E. S. Bumps, H. D. Kessler, and M. Hansen

The titanium-rich end of the Ti-Al phase diagram has been determined to the compound $TiAl_3$ (62.7 pct Al), thus joining the aluminum-rich portion previously investigated by others and completing the diagram. Micrographic analysis and X-ray diffraction were the chief methods used to determine the phase relationships.

THE Ti-Al phase diagram has been investigated as part of a program on titanium phase diagrams for the Wright Air Development Center. Included in the available information concerning the phase relationships in titanium-rich Ti-Al alloys is a brief statement by Brown[1] that: "aluminum raises the transformation temperature to 1000°C (1832°F)." This observation is interesting in that it is the first example of a metallic alloying component raising the transformation temperature of titanium, and consequently widening the field of the α solid solution. Busch and Freyer[2] presented resistivity data which indicated the formation of titanium-rich solid solutions of 1 and 3 pct Al alloys prepared by powder metallurgy methods.

Duwez[3] found that there is only one other intermediate phase other than the known compound $TiAl_3$ (62.7 pct Al). This phase, designated as γ, is of variable composition; its crystal structure is of the AuCu type and is therefore based on the composition TiAl (36.02 pct Al).

Earlier work on the constitution of the aluminum-rich alloys has been reviewed by Hansen,[4] and need not be discussed here. The tetragonal crystal lattice of $TiAl_3$ as found by Fink, Van Horn, and Budge,[5] was confirmed by Brauer.[6] Recently, Schubert[7] has reported that $TiAl_3$ is homogeneous within a limited composition range; however, the solubility limits were not given.

The Ti-Al phase diagram as presented in this paper was determined by micrographic analysis of alloys containing from 1 to 62 pct Al, annealed at and quenched from temperatures between 700° and 1400°C. The phases and phase ranges investigated extend to the compound $TiAl_3$ (62.7 pct Al) to join the aluminum-rich end of the system determined by other investigators, thus completing the diagram.

Methods of Investigation

Preparation of Alloys: Iodide titanium, 99.9+ pct pure, obtained from the New Jersey Zinc Co., and high-purity (99.99+ pct) aluminum sheet from the Aluminum Co. of America were used to make up alloy ingots weighing 10 to 20 g. Detailed descriptions of melting techniques have been presented previously for work on the Ti-Mo and Ti-Cb[8] and Ti-Si[9] phase diagrams. Briefly, the melting practice consisted of arc melting an accurately weighed charge in a copper block crucible insert in a non-consumable tungsten electrode furnace, under helium at atmospheric pressure. All ingots were carefully weighed after melting to detect possible losses. Comparison of weight before and after melting showed no appreciable weight changes. Also, chemical analyses were in close agreement with nominal compositions, Table I. The diagram as presented is, therefore, based on nominal compositions. Alloys were prepared having the following nominal aluminum contents: 1 . . . 36 (1 pct increments), 38, 40, 41, 42.5, 44, 45, 46, 47.5, 49 . . . 64 (1 pct increments), 67, 70, 80, and 90.

Table I. Nominal Composition Vs. Chemical Analysis for Typical Ti-Al Alloys

Nominal Composition, Pct Al	Chemical Analysis, Pct Al
16.0	15.84
27.3	27.08
36.0	35.89
45.0	44.96
53.0	53.16
62.7	62.97

Ingots with up to 14 pct Al were cold-pressed various amounts in order to increase the rate of attaining equilibrium structures on heat treatment. The amount of cold deformation that could be applied decreased with increasing aluminum content, until alloys containing 12 pct Al or more cracked after only a few percent deformation.

Annealing Treatments: Samples annealed at temperatures of 700° to 1000°C were sealed in

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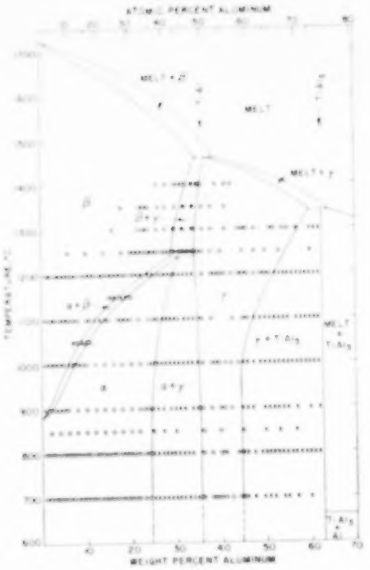

Fig. 1—Partial diagram of the Ti-Al system

evacuated Vycor bulbs. From 1000° to 1200°C, the samples were treated in sealed quartz capsules under a partial pressure of argon. Above 1200°C, an additional precaution was taken to prevent contamination of the samples by wrapping them in molybdenum sheet to avoid contact between the specimens and the quartz capsules in which they were sealed under a partial pressure of argon.

The number of alloys and the temperatures of treatment are indicated as data points in the partial phase diagram, Fig. 1. The annealing times varied from 330 hr at 700° to 48 hr at 1000°, 2 hr at 1250°, and 10 min at 1300° and 1400°C. The shorter times above 1250°C were necessary to limit the depth of surface contamination.

X-Ray Diffraction Techniques: The powders for X-ray diffraction patterns were obtained by filing

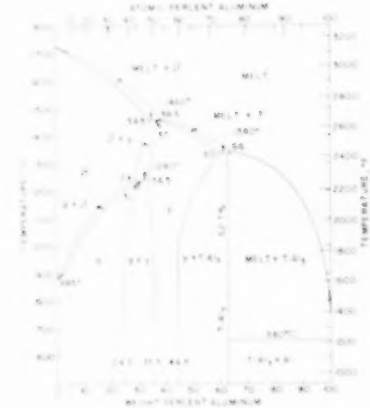

Fig. 2—Ti-Al phase diagram

or crushing annealed specimens. The powders thus obtained were sealed inside a capped titanium container which was, in turn, placed inside an evacuated Vycor capsule and heated to the prior annealing temperature for a short time. The Vycor bulbs were quenched in water, but the bulbs were not broken until the contents were cool. A standard Debye-Scherrer type camera of 7.18 cm radius, and characteristic, filtered CuK, radiation was used to obtain the patterns.

Melting Range Determinations: Solidus data were determined by incipient melting techniques.[7] First, the melting temperature for a specimen of given composition was visually observed. Then a series of specimens of the same composition were quenched at temperature intervals below the observed melting, and subsequently examined metallographically. The accuracy of the solidus data is of the order of ±15°C.

Liquidus data were obtained from cooling curves for three Ti-Al alloys. The details of the method used are covered in a previous publication on the Ti-Si system.[8]

Hardness Determinations: Vickers pyramid hardness data obtained using a load of 10 kg or more revealed considerable scatter due to irregular or deformed impressions. Fairly uniform impressions, and consequently more consistent data, were obtained with a 2½ kg load.

Discussion of Results

Phase Diagram: The essential features of the phase diagram presented in Figs. 1 and 2 are two peritectic reactions, β + melt $\rightleftarrows$ γ, and γ + melt $\rightleftarrows$ $TiAl_3$ at about 1460° and 1340°C, respectively, and one peritectoid reaction, $\beta + \gamma \rightleftarrows \alpha$, at 1240°C. The temperature of the polymorphic transformation of titanium is raised by addition of 29 pct Al from 885° to 1240°C, giving rise to extended ranges of α and β solid solutions. The solubility of aluminum in α titanium is approximately 24.5 pct at 900°C and increases to 31 pct Al at 1240°C. The intermediate phase, γ, is homogeneous over a range of composition from about 37 to 60 pct Al at the solidus temperature, and from 35.5 to 44.5 pct at 800°C. The phase relationships for the alloys higher in aluminum content than the compound $TiAl_3$ (62.7 pct Al), which are included in Fig. 2, are based on earlier work.[9]

Structural Characteristics: Microstructures of alloys quenched from the various phase fields are shown in Figs. 3 to 15.

Specimens quenched from the β field of the diagram revealed a transformation of β to serrated α, Fig. 3. The duplex structure of specimens quenched from the narrow $\alpha + \beta$ field is shown in Figs. 4 and 5. Alloys quenched from the α phase field were characterized by equiaxed grains of α solid solution, Fig. 6.

The first trace of γ appears as a precipitate of small particles along parallel striations in the α grains. At slightly higher aluminum contents, in the temperature range 700° to 1000°C, γ precipitates from the α solid solution in a lamellar fashion. This precipitate appears to have nucleated at the grain boundaries and subsequently grown into the striated α grains, Fig. 7. The amount of the lamellar structure varies from traces up to 100 pct, depending on the composition and time at the temperature of annealing. The precipitation results from the large decrease in the solid solubility of aluminum in α

Fig. 3—23 pct nominal aluminum alloy, water quenched after annealing 2 hr at 1250°C. Typical serrated α grain structure of alloys quenched from the β phase field. Etchant: 2 pct HF, 3 pct HNO_3 in water. X250.

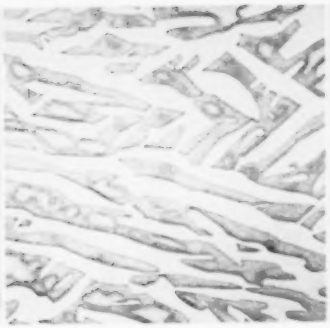

Fig. 4—7 pct nominal aluminum alloy, pressed 70 pct, water quenched after annealing 50 hr at 1000°C. Structure shows a mixture of isothermal α plus transformed β. Etchant: 2 pct HF, 3 pct HNO_3 in water. X250.

Fig. 5—25 pct nominal aluminum alloy, water quenched after 6 hr at 1200°C. Structure shows α plus transformed β. Etchant: 2 pct HF, 3 pct HNO_3 in water. X250.

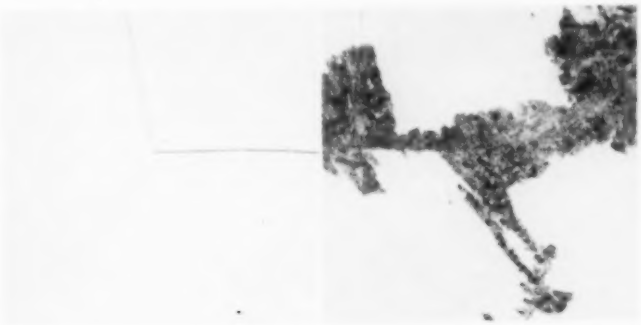

Fig. 6—8 pct nominal aluminum alloy, pressed 35 pct, water quenched after 24 hr at 1000°C. Large equiaxed grains of α solid solution. Etchant: 2 pct HF, 3 pct HNO_3 in water. X250.

Fig. 7—27.3 pct nominal aluminum alloy water quenched after 180 hr at 800°C. Structure shows nucleation and growth of a lamellar precipitate of TiAl in α. Note the striations in the α grains. Etchant: 2 pct HF, 3 pct HNO_3 in water. X150.

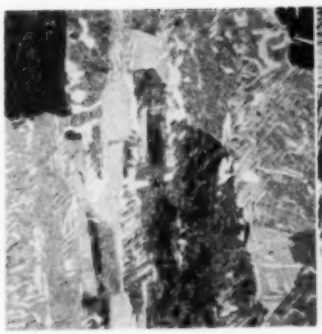

Fig. 8—34 pct nominal aluminum alloy water quenched after 6 hr at 1200°C. Structure shows a mixture of TiAl (matrix) and α. Etchant: 2 pct HF, 3 pct HNO_3 in water. X250.

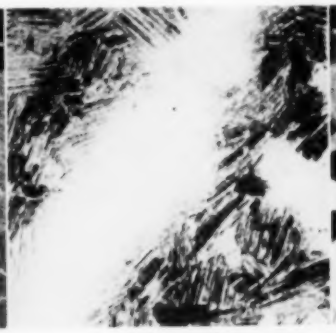

Fig. 9—32 pct nominal aluminum alloy water quenched after 10 min at 1350°C. Serrated α grain structure together with TiAl phase characteristic of alloys quenched from the $\beta + \gamma$ phase field. Etchant: 60 pct glycerine, 20 pct HNO_3, 20 pct HF. X200.

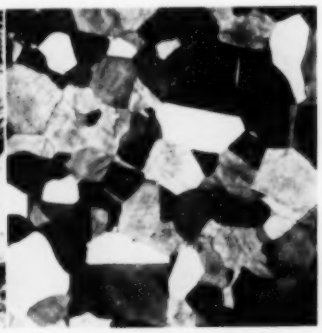

Fig. 10—36 pct nominal aluminum alloy pressed 25 pct and water quenched after 24 hr at 1000°C. Single-phase structure showing the appearance of the γ (TiAl) phase. Unetched. Polarized light. X150.

Fig. 11—49 pct nominal aluminum alloy homogenized at 1200 C, furnace cooled and water quenched after 48 hr at 1000 C. Structure shows a Widmanstatten pattern of $TiAl_3$ platelets precipitated in a γ matrix. Etchant: 60 pct glycerine, 20 pct HNO_3, 20 pct HF. Oblique illumination. X800.

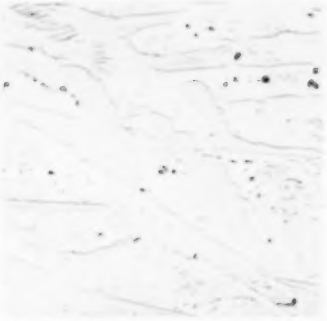

Fig. 12—61 pct nominal aluminum alloy water quenched after 24 hr at 1000 C. Structure shows large plates of primary γ phase plus Widmanstatten platelets of precipitated γ in a $TiAl_3$ matrix. Etchant: 60 pct glycerine, 20 pct HNO_3, 20 pct HF. X250.

titanium, varying from about 31 pct at 1240 C to 24.5 pct at 800 C. Massive γ appeared in alloys with an aluminum content greater than 31 pct, Fig. 8.

A typical structure of alloys quenched from the $\beta + \gamma$ field is shown in Fig. 9. As in all alloys quenched from the β field at this temperature level, the white phase appears serrated due to the transformation $\beta \rightarrow \alpha$ during the quench. The γ phase has precipitated in parallel plates along the boundaries of the original β grains.

The structure of the γ phase at 36 pct Al is shown in Fig. 10. As depicted in Fig. 1, this phase is homogeneous over a wide range of composition. Alloys with higher aluminum contents approaching the $\gamma/\gamma + TiAl_3$ boundary become increasingly brittle and contain many cracks, but the structure of these alloys after annealing treatments shows the same response to polarized light as does the 36 pct Al alloy of Fig. 10. In the as-cast condition, these alloys were cored. Although homogenization anneals for as long as 24 hr at 1200 C almost completely eliminated coring, traces of residual inhomogeneity within the grains caused some difficulty in accurately locating the $\gamma/\gamma + TiAl_3$ boundary. Longer homogenization times could possibly have eliminated this small amount of residual coring; however, longer exposure times at these high temperatures were considered impractical. The resulting scatter in data limits the accuracy of locating the $\gamma/\gamma + TiAl_3$ boundary to about ±1 pct.

Fig. 11 illustrates the structure of a homogenized 49 pct Al alloy quenched after 48 hr at 1000 C and shows a Widmanstatten pattern of $TiAl_3$ platelets, which precipitated from the saturated γ phase. Given the same treatment, alloys containing 56 to 59 pct Al revealed a similar Widmanstatten precipitation of $TiAl_3$ in a γ matrix with the addition of massive $TiAl_3$ globules. The latter were apparently formed by the peritectic reaction γ + melt $\rightarrow TiAl_3$, due to the rapid solidification of the ingot, and were not fully absorbed by the γ phase during the subsequent homogenization anneal.

Fig. 12 illustrates the structure of a 61 pct Al alloy quenched from 1000 C and shows primary plate-like crystals of the γ phase in a matrix of $TiAl_3$. The Widmanstatten pattern in the $TiAl_3$ matrix developed during the anneal, and represents γ platelets precipitated from the saturated $TiAl_3$ phase. This proves that $TiAl_3$ has a certain homogeneity range which becomes narrower with fall in temperature.

The $TiAl_3$ phase is formed by the peritectic reaction Melt + $\gamma \rightarrow TiAl_3$. Evidence of this is given in the as-cast structure of alloys with 61 and 62 pct Al, Figs. 13 and 14. Fig. 14 exhibits primary γ plates

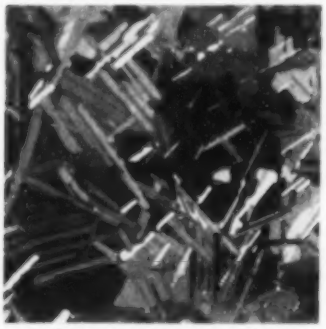

Fig. 13—61 pct nominal aluminum alloy as-cast. Structure shows primary plates of γ in a $TiAl_3$ matrix giving evidence that $TiAl_3$ was formed by a peritectic reaction. Unetched. Polarized light. X100.

Table II. Lattice Parameters of the Alpha Ti-Al Phase

Wt Pct Al	Atomic Pct Al	c (Å)	a (Å)	c/a	Temperature Quenched from, °C
0	0	4.679	2.943	1.591	850
4	6.88	4.668	2.929	1.594	850
7	11.81	4.670	2.923	1.598	1000
9	14.95	4.661	2.924	1.594	1000
10	16.49	4.661	2.912	1.601	850
14	23.27	4.633	2.892	1.602	850
22	33.39	4.619	2.882	1.603	850
25	37.15	4.614	2.881	1.602	850
29	42.94	4.617	2.878	1.604	1200

Table III. Lattice Parameters of the Gamma Ti-Al Phase

Wt Pct Al	Atomic Pct Al	c (Å)	a (Å)	c/a	Temperature Quenched from, °C
49	52.13	4.065	3.984	1.020	1200
61	55.23	4.067	3.972	1.024	1200
56	60.22	4.083	3.967	1.030	1200
55	68.47	4.089	3.949	1.035	1200

Fig. 14—62 pct nominal aluminum alloy as-cast. Structure shows two γ plates surrounded by peritectically formed $TiAl_3$ which in turn is surrounded by low melting aluminum in the interstices. Etchant: 60 pct glycerine, 20 pct HNO_3, 20 pct HF. X250.

Fig. 15—64 pct nominal aluminum alloy as-cast. Structure shows primary $TiAl_3$ surrounded by aluminum in the grain interstices. Etchant: 60 pct glycerine, 20 pct HNO_3, 20 pct HF. X250.

surrounded by peritectically formed $TiAl_3$, which in turn is surrounded by the low melting aluminum phase seen in the fine interstices of the $TiAl_3$ crystals. The positive identification of the γ and $TiAl_3$ phases was aided by their highly different characteristics when examined under polarized light. At aluminum contents higher than that of the $TiAl_3$ phase, the structure, as shown in Fig. 15, reveals primary $TiAl_3$ phase surrounded by low melting aluminum.

X-Ray Diffraction Results: X-ray diffraction studies were made on a series of α solid solution alloys, and also on a series of single-phase γ alloys, to determine the lattice parameters and the solubility limits of aluminum in the α and γ phases. It was also desired to determine whether the β to α transformation on quenching alloys from the β field could be suppressed.

A summary of the lattice parameter measurements for the α phase is given in Table II. A graphic presentation of the effect of aluminum additions on the parameters of the hexagonal close-packed lattice, as shown in Fig. 16, reveals the very pronounced effect of initial additions of aluminum on the dimensions of the titanium lattice. Near the solubility limit, the curves appear to flatten off greatly. The markedly reduced slope of the parameter curves makes a parametric determination of the α solubility limit impractical. The X-ray results also confirmed the metallographic observation that the β phase is not retained by rapid quenching.

The crystal structure of the γ phase between 38 and 55 wt pct Al is ordered face-centered tetragonal, with an axial ratio close to unity. A summary of the lattice parameter measurements is presented in Table III, and a graphic representation appears in Fig. 17. The lattice parameters are linearly dependent upon, and show increasing tetragonality with, increasing percentages of aluminum. These alloys have been quenched from 1200°C, indicating that either the ordered structure is stable to high temperatures, or the disorder ⇌ order transformation, if any, cannot be suppressed by quenching.

Melting Range Determinations: The data shown in Fig. 18 were obtained by incipient melting and thermal analysis methods described earlier. The solidus curve shows that aluminum additions lower the melting point of titanium to approximately 1460°C at 34.5 pct Al. From 38 to 60 pct Al, a change in slope of the solidus curve occurs, which is in excellent agreement with the liquidus points of alloys with 39, 45, and 50 pct Al, determined by thermal analysis, Fig. 18. All the evidence indicates that there is a peritectic reaction: β + melt ⇌ γ at a temperature close to 1460°C between approximately 34.5 and 38.5 pct Al. This correlates very well with an extrapolation of the boundaries of the β + γ field as shown in Fig. 1.

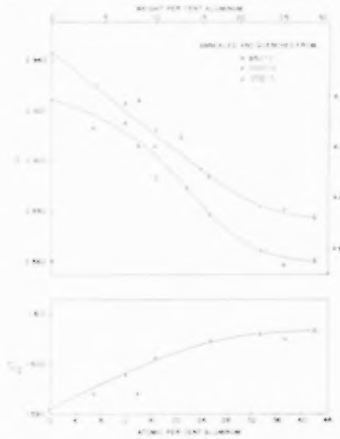

Fig. 16 (left)—Lattice parameter-composition curves for alpha solid solution Ti-Al alloys. The ordinates are in Angstrom units.

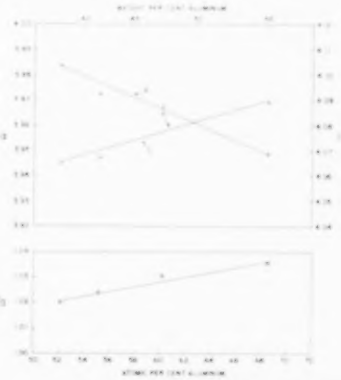

Fig. 17 (right)—Lattice parameter-composition curves for gamma solid solution Ti-Al alloys, annealed and quenched from 1200°C. The ordinates are in Angstrom units.

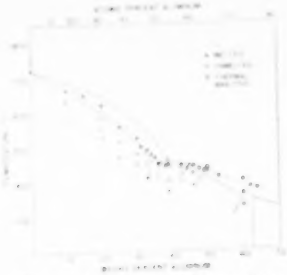

Fig. 18—Melting ranges for the Ti-Al system.

Thermal analysis did not give a definite indication of the width of the melting range for the three alloys tested (39, 45, and 50 pct Al), but together with incipient melting data the range appears to be fairly narrow for the compositions investigated.

Some scatter of the melting temperature was encountered in determining the melting points of the 60 to 64 pct Al alloys, because of the presence of low melting aluminum phase formed by the incomplete peritectic reaction. The peritectic temperature of 1340°C shown in Fig. 1 is lower than the 1355°C melting point of $TiAl$ reported by Manchot and Leber.[7] These authors did not investigate alloys with lower aluminum contents than $TiAl_3$ and consequently failed to observe a peritectic reaction. On the other hand, the present data are based on the extrapolation of the $\gamma/\gamma + TiAl$ boundary, together with microscopically observed evidence of a peritectic structure.

Hardness Test Results. Vickers hardness data for as-cast alloys and those heat-treated at 800° and 1200°C are presented graphically in Fig. 19. The data include alloys from 0 to 90 pct Al.

Aluminum is a very effective hardener of α titanium. The hardness curves show an increase in hardness in the 1 to 25 pct Al α solid-solution range. A maximum hardness peak is reached at 25 or 30 pct Al, depending on the treatment. This composition range exhibited the most scatter in hardness data. It appeared that most of the scatter was due to the brittle characteristics of the alloys in this range of composition.

Alloys containing 25 to 35 pct Al, which structurally revealed an increased amount of γ phase, showed decreasing hardness, reaching a minimum value for the 36 pct Al alloy. This alloy has been identified metallographically as a single-phase, γ.

Hardness in the 40 to 60 pct Al composition range increases to a second maximum in the hardness curve, which occurs at $TiAl_3$ (62.7 pct Al).

Hardness data for alloys higher than 60 pct Al were restricted to as-cast alloys. The hardness drops sharply from 62 to 90 pct Al and levels off to approximately the hardness of pure aluminum.

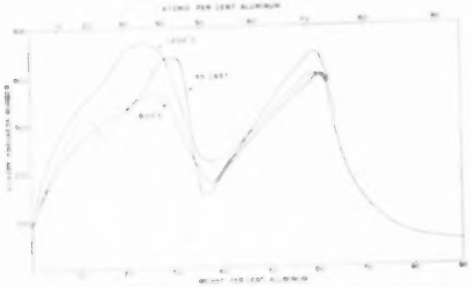

Fig. 19—Comparative Vickers hardness composition curves for Ti-Al alloys treated as indicated.

Summary

The Ti-Al phase diagram was determined with iodide titanium-high purity aluminum are melted alloys from 0 to 64 wt pct Al. The principal features of the diagrams are as follows:

1—A peritectic reaction, β + melt $\rightleftarrows \gamma$, between 34.5 and 38.5 pct Al at approximately 1460°C.

2—A second peritectic reaction, γ + melt $\rightleftarrows TiAl_3$, between 60 and 64 pct Al at 1340°C.

3—A peritectoid reaction, $\beta + \gamma \rightleftarrows \alpha$, between 29 and 34.5 pct Al at approximately 1240°C.

4—The $\alpha \rightleftarrows \beta$ transformation temperature is raised from 885°C at 0 pct Al to 1240°C at 29 pct Al. A narrow $\alpha + \beta$ field is indicated. The β phase transforms to serrated α on quenching.

5—The solubility limit of aluminum in α titanium is 24.5 pct at 900°C. The maximum solubility of aluminum in β titanium is 34.5 pct at 1460°C.

6—The lattice parameters of the solid solution change considerably with initial additions of aluminum and level off near the solubility limit. X-ray diffraction patterns confirmed the metallographic observation that β is not retained by rapid quenching.

7—The γ phase is of variable composition, from 37 to 60 pct Al in the solidus and from 35.5 to about 44.5 pct Al at 800°C.

8—The γ phase has an ordered face-centered tetragonal lattice, with an axial ratio close to unity, and is based on the composition $TiAl$. Increasing aluminum content increases the axial ratio slightly.

9—The hardness of α solid solution alloys increases with the addition of aluminum to titanium. The γ phase has a minimum hardness at 36 pct Al, which increases with further additions of aluminum, reaching a maximum at the composition $TiAl_3$.

Acknowledgments

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Compression Texture of Iodide Titanium

by D. N. Williams and D. S. Eppelsheimer

The compression texture of iodide titanium is determined and found to consist of a [0001] texture rotated 15° to 30° from the axis of compression. As the amount of reduction increases, the angle of rotation decreases. The 0001 rotation showed a slight preference for a <1010> rotation axis.

RECENT investigation of the cold rolled texture of titanium[1] has shown this metal to exhibit a new type of rolling texture. This texture can best be described as a (0001) [10$\bar{1}$0] with the 0001 planes rotated approximately 30° to the transverse direction around a <1010> rotation axis. Rather good results in explaining the cold rolled texture have been obtained by other workers[2] by examination of the tension and compression textures.

No information was found giving the tension texture of titanium. Generally the tension texture of a hexagonal close-packed metal is identical with its wire texture. The drawn texture of titanium has been investigated[3] and has been found to be predominately [10$\bar{1}$0]. Since this is the common tension texture of hexagonal metals of low c/a ratio, the explanation of the difference between the rolling texture of titanium and the rolling texture of other hexagonal metals is not indicated by examination of the tension texture. Therefore, the compression texture of titanium should be particularly important in the explanation of the unusual transverse rotation of the 0001 planes found in the cold rolled texture.

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The compression texture of titanium was examined by Yen.[2] His results showed titanium to exhibit the usual [0001] texture up to 85 pct reduction. At higher reduction a double texture appeared in which the 0001 planes were rotated from 10° to 30° from the plane of compression. This change in the compression texture seemed unusual since the tendency for hexagonal close-packed metals is to approach the [0001] texture as the idea compression texture. Mechanical twinning generally occurs over a considerable range of reductions beginning at a rather low reduction.

The samples used by Yen in his investigations were prepared by two methods. Compression in a testing machine was used up to a reduction of 85 pct. Compression by compression rolling was used at higher reductions. Since the change in texture occurred at the same reduction as the change in the method of compression, a connection between the two seemed likely. To check this possibility and to locate the position of the change in texture definitely, if it was found to exist, the compression texture of titanium was re-examined.

An iodide titanium crystal bar (99.9 pct Ti) produced by the New Jersey Zinc Co. and donated by the Titanium Alloy Manufacturing Div. of the National Lead Co. was used in this investigation.

Procedure

The investigation of the compression texture was carried out in three steps: compression, specimen

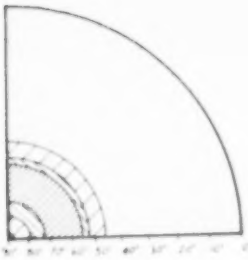

Fig. 1—0001 pole figure of compressed iodide titanium. 24.1 pct reduction.

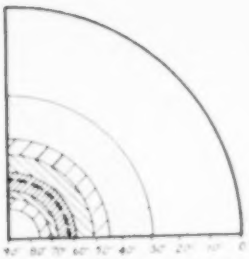

Fig. 3—0001 pole figure of compressed iodide titanium. 80.1 pct reduction.

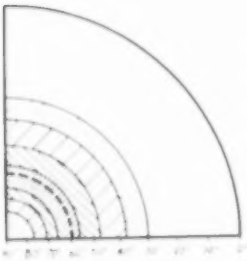

Fig. 2—0001 pole figure of compressed rolled iodide titanium. 50 pct reduction.

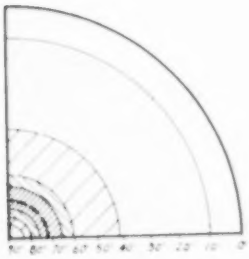

Fig. 4—0001 pole figure of compressed rolled iodide titanium. 98.9 pct reduction.

preparation and X-ray examination, and correction and plotting of the X-ray data.

Compression: Samples were cut directly from the iodide titanium crystal to a thickness varying from 0.60 to 0.25 cm, depending on the required final reduction. The ends of the sample were made parallel by alternate etching and light polishing. The samples were either compressed between lubricated plates or compression rolled in a set of 2 in. laboratory rolls.

An Olsen testing machine of 60,000 lb capacity was used to compress those samples subjected to compression between lubricated plates. During the compression process the samples were frequently removed from the machine and re-lubricated. The removal of samples was made necessary by the low load limit of the machine which required that the area be frequently reduced before further compression could be obtained. The approach to uniaxial compression was better than expected. The external shape of the sample after compression showed rather uniform lateral spread. The possibility of secondary stresses cannot be overlooked, however.

Several samples were reduced entirely by compression rolling. A small reduction per pass was used with a clockwise rotation of from 3° to 5° after each reduction. All samples were rotated completely around the compression axis at least twice. The compression-rolled samples showed a circular section after rolling indicating uniform compression.

By selecting the reductions by the two methods so that the ranges of compression overlapped (see Table I), any change in texture due to change in compression method would be apparent. The cone compression test, probably the most reliable method of obtaining pure compression, was not used due to the necessity of obtaining thin, parallel-sided samples for X-ray examination.

The crystalline nature of the iodide titanium bar was such that the outer third of the bar separated at the crystal boundaries during compression. Therefore all X-ray specimens were taken from the center part of the compressed sample. Although this separation was undesirable, remelting the bar in the facilities available would have caused contamination of the metal.

X-ray Examination: X-ray examination of the samples was made with a North American Philips X-ray Goniometer Unit using CuKα radiation. The Schulz-Decker method of pole figure determination was used.[7] Two X-ray specimens are required using this method. Transmission specimens of approxi-

Table I. Effect of Reduction on the Compression Texture of Titanium

Pct Reduction	Compression Method	Angle Between (0001) Maximum and Compression Axis	Relative Intensity of (0001) Maximum
24.1	Compressed	32.5° to 25°	20*
54.1	Compressed	20°	100†
50.0	Compression rolled	26°	35
80.1	Compressed	27.5°	40
98.2	Compressed	25°	35
98.9	Compression rolled	24°	42
99.2	Compression rolled	20°	39
99.9	Compression rolled	13.5°	33

* From deformed material (est).
† Probably in error (see text).

mately 0.003 cm and reflection specimens of approximately 0.020 cm or greater were prepared by etching in a HF, HNO_3, H_2O solution (1:2:5). Uneven etching and pitting was a problem, particularly with samples of low reduction. In all cases at least one third of the initial thickness was removed by etching to prevent surface effects.

Patterns were made from 10° to −50° using the transmission method and from 25° to −25° using the reflection method. The region between −50° and −25° overlapped and served as a means of correlating the two patterns. Readings were made along from two to four diameters of the pole figure, depending upon the agreement found. Because of the high absorption coefficient of titanium, an absorption correction was necessary for all transmission intensity readings.

Intensity readings from the 1010, 0001, and 1011 planes were measured for each sample. Readings were taken at 2½° intervals along the diameter.

Plotting of the Pole Figures. The data taken from X-ray patterns can be plotted directly over a polar stereographic net after intensity correction of the transmission readings. The plane of the pole figure corresponds to the plane of compression. The axis of compression is perpendicular to the plane of the pole figure. Thus the pole figure consists of a series of concentric intensity rings centered on the axis of compression.

Only one quadrant of the pole figure is plotted. Data from readings in other quadrants were averaged with the data from the quadrant plotted. The pole figures for the 1010 and 1011 planes were used as a check on the accuracy of the 0001 pole figure. Only the 0001 pole figures are included in the paper. The 1010 and 1011 pole figures supported the 0001 pole figure very well and also showed a slight degree of preference for a <1010> axis of rotation.

After correction of the transmission patterns for absorption, the readings were compared with those obtained from the reflection patterns in the overlapping region, and a uniform correction was applied to make them correspond. The final readings were then reduced so that all readings fell within the range of 0 to 50 intensity units, the maximum having an intensity reading of 50 units. These readings were plotted in the pole figure using five degrees of intensity as follows: 0 to 10 units, blank; 10 to 20 units, dotted; 20 to 30 units, wide lining; 30 to 40 units, medium lining; and 40 to 50 units, narrow lining. The position of the maximum reading is shown by a dashed line when a definite maximum exists.

To determine the relative strength of each maximum reading, the intensity distribution of each sample was adjusted to give a constant total intensity over the range examined. The maximum intensity was then measured for comparison with that of the other samples.

Experimental Results

Four of the 0001 pole figures found for compressed titanium are given in Figs. 1 to 4. The texture produced by compression cannot be distinguished from that produced by compression rolling. The general appearance of the texture is the same in each case. This texture can be described as a rotation of the 0001 planes from 15° to 30° away from the plane of compression. Examination of the pole figures shows that as the reduction increases the angle of rotation decreases. The angle between the 0001 maximum and the compression axis for each reduction is given in Table I. The maximum intensity occurs at an angle of rotation of 30° at low reductions and at an angle of rotation of 17.5° at 98.9 pct reduction. No definite maximum existed in the sample compressed 24.1 pct.

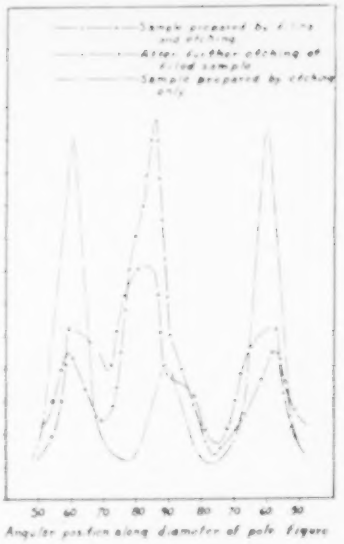
Sample prepared by filing and etching
After further etching of filed sample
Sample prepared by etching only
Intensity of 0002 diffracted rays
50 60 70 80 90 80 70 60 50
Angular position along diameter of pole figure

Fig. 5—Comparison of 0001 patterns of compressed titanium (34.3 pct reduction) after various methods of specimen preparation.

A second noticeable feature of these pole figures is the low intensity occurring at the center of the pole figure. This region corresponds to the position of the [0001] texture which is usually considered as the ideal compression texture for a hexagonal close-packed metal. The low intensity of this region indicates that some type of twinning is removing the 0001 poles from the ideal position.

The method used in plotting the pole figures in this paper gives no indication of the relative intensity of the maxima of the samples. Corrected maximum intensities were calculated and are shown in Table I.

The figure given in Table I for the relative intensity of the maximum of the sample compressed to 24.1 pct reduction is not strictly comparable with those from the other samples. This sample showed two distinct patterns. The first, which was used in plotting the pole figure given in Fig. 1, arose from the deformed or fragmented structure of the metal. A second pattern, arising from a large block of relatively undeformed metal, was not included in the pole figure. This pattern showed the usual sharp, strong intensity readings of a single grain area. Only the intensity readings from the first pattern were used in correcting the intensity distribution for a constant total intensity.

Fig. 5 shows the 0001 intensity curves of the sample compressed to 34.3 pct reduction. The X-ray specimens of this sample were prepared by three methods. The original sample after compression had a thickness of approximately 0.35 cm. To reduce the

opportunity for pitting during etching, the sample was filed to 0.15 cm and then reduced to 0.08 cm by alternate etching and light polishing. The X-ray examination of this sample showed the usual double texture plus a strong [0001] texture. The [0001] maximum did not occur at 90° indicating that it may have been a secondary texture produced by the filing operation. The sample was etched further to 0.065 cm and again examined. The unsymmetrical central maximum was much lower with a corresponding increase in the double maxima. A third specimen was prepared by etching alone. The readings obtained with this specimen are seen to be considerably different from those obtained using the previous methods of specimen preparation. The double texture is very pronounced with no maximum occurring at 85°. A small [0001] maximum is seen at 90°.

Discussion of Results

The appearance of a uniform texture over the complete range examined indicates that compression between parallel plates and compression rolling produce similar results, as was found by Barrett[7] in the case of iron.

The compression texture of titanium is seen by the results shown in Fig. 5 to be sensitive to a shear stress parallel to the plane of compression. The effect of this stress is noted at a considerable distance into the sample. The [0001] texture reported by Yen may possibly have been a result of this effect.

The results obtained from the examination of the sample compressed 34.3 pct are not reliable. The degree of preferred orientation of this sample is considerably higher than that of the other samples, as is shown in Table I. This difference is believed due to some deformation introduced into the sample during its original preparation. Although it shows the usual double maxima it also has a distinct [0001] maximum (see Fig. 5) which was absent in all other samples.

If the corrected intensity of the other seven samples is compared, it is seen that the compression texture soon reaches a more or less stable intensity and that the only change brought about by increased reduction is a shift in the position of the 0001 maxima. This suggests that the observed texture is an equilibrium texture existing between the stable endpoints of slip and twinning. A movement of the boundary between the slip and twinning regions due to unequal changes of the critical shear stresses would then account for the shift in the position of the 0001 maxima.

According to a recent analysis of the mechanics of twinning by Barrett,[7] twinning on the 1012 planes is not possible in close-packed hexagonal metals with a c/a ratio less than 1.732 if the axis of the compression is along the hexagonal axis of the lattice. In titanium with a c/a ratio of 1.587, 1012 twinning, the usual type of hexagonal twinning, could not be the means by which the central low intensity region is kept depleted of 0001 planes. Since rotation by slip on the base planes should be continually moving 0001 planes into this region, an explanation of its low intensity is required.

A recent investigation of the slip and twinning elements of titanium[8] has shown titanium to twin on the 1012, 1122, and 1121 planes. Both 1122 and 1121 twinning are able to twin in compression from a [0001] position and therefore to remove 0001 planes from the center of the pole figures in this paper.

Examination of the $10\bar{1}0$ and $10\bar{1}1$ pole figures of the samples showed a slight tendency for the 0001 rotation to occur about a $<10\bar{1}0>$ axis. This type of preferred orientation would result from $\{10\bar{1}1\}$ $<11\bar{2}0>$ slip. The lack of any appreciable 0001 intensity along the circumference of the 0001 pole figures indicates that in compression $\{10\bar{1}0\}$ $<11\bar{2}0>$ slip is relatively unimportant. Both of these modes of slip have been reported for titanium.[9]

If cold rolling is considered to be approximated by tension in the rolling direction and compression along the rolling plane normal, a combination of the compression texture reported here at lower reductions and of the drawn texture reported by Yen would produce a cold rolled texture which would agree closely with that determined by Clark.[1]

Summary

1—Compression samples with eight different reductions were prepared and the 0001, 1010, and 1011 pole figures determined.

2—A rotation of the base planes 30° from the compression plane was found in the initial reductions. The amount of rotation of the base plane from the compression plane decreased with increased reduction. A minimum angle of rotation of 17.5° was found at a reduction of 98.9 pct.

3—Compression in a testing machine and by compression rolling were found to produce similar results.

4—The compression texture was found to be considerably changed by any shearing stress parallel to the plane of compression. This stress tended to produce a [0001] texture.

5—An equilibrium type texture developed by interaction of $\{0001\}$ $<11\bar{2}0>$ and $\{10\bar{1}1\}$ $<11\bar{2}0>$ slip and of 1012, 1122, and 1121 twinning is suggested as a possible explanation of the compression texture of titanium.

Acknowledgments

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Observations of Creep of the Grain Boundary In High Purity Aluminum

by H. C. Chang and N. J. Grant

CREEP studies and measurements in most instances are based on a relatively gross gage length. Even in some recent theoretical studies on the mechanism of creep, changes were followed by means of X-rays and metallography without regard for the behavior of the individual grains and grain boundaries.[1-3] Analytical studies of creep have frequently indicated that more than one competing process is operative and that the ultimate creep performance depends on the effects of temperature, time, and strain rate as well as grain size.[4-6] The grains and grain boundaries are known to respond differently to these variables but the extent of the response is still little known, especially in polycrystalline materials where the relative orientations of grains and grain boundaries are important.

This paper reports some experimental observations of the behavior of the grain boundaries and their effect on the deformation in the grains. High purity aluminum was used to avoid complications of aging reactions, second phase, inclusions, etc.

Experimental Procedure

A small vertical nichrome wound furnace was built to permit direct loading on the test specimen. This furnace is capable of operating up to about 1300 F with reasonable temperature distribution. A ground rectangular quartz window, 1.25x0.5 in., is imbedded in the external stainless steel tube to permit observations of the creep process with a microscope. An argon atmosphere was provided to prevent oxidation. The quartz window caused a temperature gradient between the back and front surfaces of the specimen of 15° at 700°F. The temperature gradient could be cut down to 3°F along the length of the specimen by adjusting shunts across the furnace windings.

An ordinary metallurgical microscope with a working distance of 14 mm was rebuilt so that it could be moved in three mutual perpendicular directions. Visual examination could be made with either a 30X eyepiece or a 15X filar eyepiece. Using an objective of focal length 24.3 mm and a numerical aperture 0.20 a total magnification of 120X or 240X could be obtained.

High purity aluminum rated at 99.995 pct was supplied by the Aluminum Co. of America as $\frac{5}{8}$ in. rod.* It was first machined to $\frac{1}{4}$ in. diam rod, and then the gage portion was further machined to 3/16 in. diam. The ends were $\frac{1}{4}$ in. diam and threaded to fit available specimen holders. Two opposite flat surfaces of the gage portion were obtained by milling, giving final dimensions of the gage portion of 3/4x3/16x3/32 in.

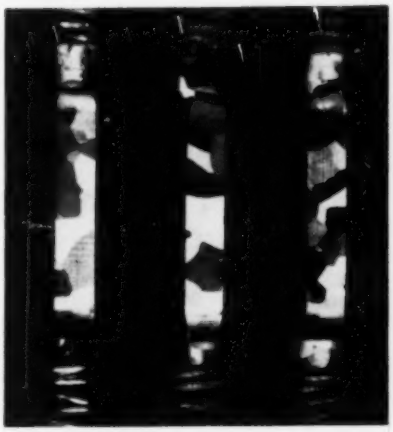

Fig. 1—Macrostructures and dimensions of specimens. Etched by Tucker's reagent. X1.8

* Analysis of the high purity aluminum was: Cu, 0.0017 pct; Fe, 0.002 pct; Si, 0.001 pct; Mg, 0.0002 pct; and Ca, Na, 0.0008 pct.

Machined specimens were first electropolished in Jacquet's solution.[7] Reference marks were produced by pressing a thin sewing needle into the surface of the specimen. The specimen was then annealed at 900°F for 24 hr and at 1150°F for 12 hr. Typical macrostructures of specimens treated in this manner are shown in Fig. 1. The range of the grain size was 1 to 5.5 mm (see Fig. 1), but most of the grains were of 2.5 mm and went through the whole thickness of the specimen. The specimen was re-electropolished and etched electrolytically to reveal the grain boundaries. In order to reveal the orientation difference of the grains, etch pits were produced on the surface of the grains by etching in a mixture of hydrochloric and nitric acids (HCl, 45 pct; HNO_3, 10 pct; H_2O, 45 pct, by volume).

The specimens were very carefully put into the holders and furnace; yet even with this care, fine slip lines were developed on the specimen surface.

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Later it was found that, in addition to the etch pits, these slip lines were of great help in determining the orientation of grains and in evaluating the behavior of grain boundaries. The load needed for this experiment was only 1.5 lb (stress about 85 psi). In order not to disturb the specimen by loading when it was very weak at the creep temperature, direct load was applied at room temperature and the specimen was brought up to temperature under load.

The needle points produced in the surface of the specimen gave very bright and sharp points under the macroscope and therefore served well as reference marks for creep measurement. Measurement could be made either by a filar eyepiece micrometer or by a dial gage. The accuracy of measurement depended on the size and the constancy of reference marks on the surface of the specimen.

With creep strain up to about 50 pct, no difficulty was encountered in measurement because the sharpness of the reference marks was maintained and their shapes did not change very much. The size of the reference marks was about 0.01 mm in diam and the distance between two reference marks was about 0.6 mm; thus the error of strain measurement should not be greater than 1.5 pct.

Results

The results presented below came from creep studies on two specimens, numbered P-3 and P-8. Both specimens were tested in creep at 700°F under a stress of 85 psi, but the duration of creep for each specimen was different as was the creep strain. Neither specimen was tested to rupture. The average creep strain of the whole gage length and the duration of creep of specimen P-3 were 2.5 pct and 1.5 hr, respectively, and those of P-8, 7.7 pct and 81 hr, respectively. Specimen numbers have been included after the figure numbers in this paper. All the micrographs were taken after the specimen was cooled down to room temperature with the load still on. Over 100 micrographs were taken in different parts of these two specimens, both on deformed and repolished surfaces. Only the most significant and interesting ones can be presented here for the sake of brevity. All the micrographs are so placed in the paper that the direction of the applied tension runs from top to bottom. It should be emphasized that all the grain boundaries, almost without exception, were fairly straight after the above-stated annealing treatments.

Boundary Slip and Migration

It can be seen from Fig. 2a that the grain boundary in the center of the field underwent successively alternating sliding and migration. The positions of the initial and final grain boundaries can be judged by the appearance of etch pits and are also indicated by letters *I* and *F*, representing the initial and final grain boundaries respectively. Since the etch pits were put in before creep, the initial grain boundary should be the demarkation line of

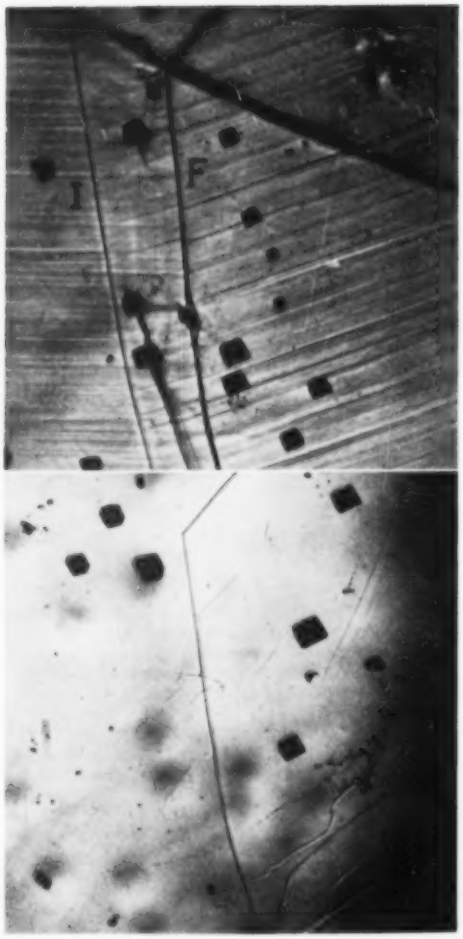


Fig. 2a (top)—Specimen P-3. Deformed surface showing the steps of grain boundary migration and sliding during creep. Etch pits put in before test. *I* and *F* indicate the initial and final grain boundaries, respectively. Slanting line that crosses *I* and *F* is fold line. X150.

Fig. 2b (bottom)—Specimen P-3. Same field as Fig. 2a repolished and etched. Initial grain boundary and fold (or band) disappeared. Etch pits put in after test. Crater-like regions are etch pit locations seen in Fig. 2a. X150.

Fig. 3—Specimen P-8. Creep deformed surface showing the grain boundary attracting effect of the needle point indentation and the subgrain structure in the bottom grain. X50.

the two differently shaped etch pits corresponding to the two grains. The position of the final boundary was also proved by repolishing and etching the specimen whereby the initial boundary disappeared and the final boundary remained (see Fig. 2b). Evidence of successively alternating and cooperative boundary sliding and migration can also be obtained by observing the course of the slip lines of the right grain which were produced during handling in putting the specimen into the furnace (shown in Fig. 2a). The original straight slip lines are now sharply displaced in the field between the initial and final grain boundaries due to the grain boundary sliding and migration. It is necessary to point out that the slip lines of the left grain are propagated continuously across the migrated zone without being bent. The fact that they were not bent indicated that these slip lines must have been developed at the conclusion of boundary migration, by handling of the specimen after test.

It is interesting to note that the course of grain boundary migration was affected by the existence of etch pits. This effect persisted quite deeply into the thickness of the specimen. The same effect still showed up even after removing the etch pits by electropolishing off a surface layer of about 0.02 mm. Reference marks produced by needle points and scratches exerted the same effect on boundary migration, as shown in Fig. 3.

Fig. 4 shows a creep curve obtained between the two needle holes across the grain boundary of Fig. 5a, a grain boundary which has undergone successively alternating and cooperative sliding and migration during the creep test. The steps of grain boundary sliding and migration are more clearly shown in Fig. 6 around the lower triple intersection point of Fig. 5a, now at a magnification of 150X.

In comparing Figs. 5a and b (the latter showing the repolished and etched surface), the positions of the initial and final boundaries are readily noted. The crater-like spots in Fig. 5b are the old etch pits of Fig. 5a which were put in prior to loading. Note that they do not coincide with the new etch pits after repolishing. This statement also applies to other micrographs of repolished surfaces (Figs. 2b, 9b, and 10b).

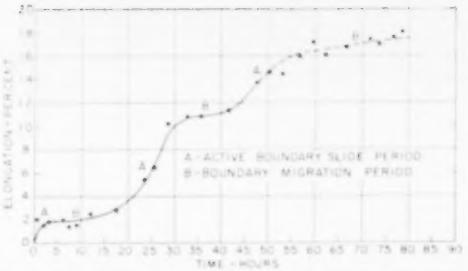


Fig. 4—Specimen P-8. Creep curve between two reference marks across a grain boundary, see Fig. 5a.

Two other items are of considerable interest. The extent of grain boundary sliding is evidenced by the thickness of the shadow which is cast by an adjoining grain. The amount of grain boundary sliding is thus shown in Fig. 5a, this shadow still being in evidence in Fig. 5b after repolishing the surface. Second, note the external surface evidence of what is very probably subgrain formation in Fig. 5a where the light effects are ideal.

It was observed with the high temperature microscope that boundary sliding and migration were carried out in successive steps as a function of time. The experimental curve shown in Fig. 4 for the two pin points across the grain boundary of Fig. 5a substantiates this. The period during which active boundary sliding takes place is characterized by the fact that the creep rate increases to rather high creep rates before the period of grain boundary migration takes place, resulting in a decreasing creep rate. The period of grain boundary sliding will be called "active boundary sliding period," while the latter period will be called "boundary migration period." After a period of time, boundary migration gives way again to active boundary sliding, resulting again in an increased creep rate.

It should be emphasized that in high temperature creep a grain boundary situated in a favorable position for sliding, slides from the very beginning of the creep test and constitutes essentially all of the noted primary creep, while the interior of the grain

Fig. 5a—Specimen P-8. Deformed surface showing boundary sliding and migration around the triple points. Thickness of the shadows produced by grain boundary sliding suggests the relative amount of sliding. Note the subgrain formation in the top left field. X50.

Fig. 5b—Specimen P-8. Same field as Fig. 5a repolished and etched. Initial grain boundary disappeared; final grain boundary remained. Crater-like regions are etch pit locations seen in Fig. 5a. X50.

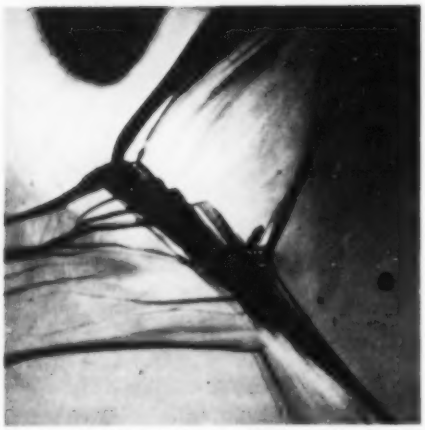


Fig. 6—Specimen P-8. Higher magnification of the lower triple point of Fig. 5a showing more clearly the steps of boundary sliding and migration. X150

does not show any appreciable deformation initially. This was proved both by visual observations and strain measurements. This phenomenon is not in accord with the accepted mode of deformation during primary creep, that is that slip takes place along crystallographic planes in the grains.

Effect of Boundary Sliding on the Deformation of Grains

Two main types of deformation of grains around a triple point, the point at which three grain boundaries meet, were observed. Fairly straight "folds" or bands may be developed in a grain and appear as extensions of the grain boundary which lies about 180° in the opposite direction. This is shown in Fig. 7a. The width of the "folds" in the grain was observed to increase with the width of the opposite grain boundary. It can be seen from Fig. 7a that the traces of these "folds" on the grain surface did not coincide with the traces of pre-existing slip lines and that they tended to stop and deviate as they entered into the grain. The same kind of "fold" is shown even more strikingly in Fig. 8. In this case, the traces of pre-existing slip lines happen to make large angles with the trace of the fold, permitting a clear picture of the appearance of the fold. The pre-existing slip lines were rather sharply and uniformly bent through the fold. This fold could not be revealed again after polishing, this being true of other such folds (see also Figs. 2b and 7b). Slip lines reproduced by small amounts of deformation at room temperature after repolishing were shown to go through the position of the fold without appreciable bending. Note from Fig. 8 how the other two initial grain boundaries at the triple point in the top center of the field were displaced by heavy sliding and migration along the third boundary, and also how the existence of etch pits caused the migrating boundary to be very wavy.

The second type of deformation in the grain caused by boundary sliding had a ridge-and-valley appearance (Fig. 9a). These areas were made up of a series of such valleys and ridges which propagated into the grain away from the boundary as the amount of boundary sliding increased. The same type of deformation shown in Fig. 10a took place not only around the corner of the triple point but also along both sides of the heavily slid boundary. As a matter of fact, the total creep strain (from Fig. 10a) measured between two reference marks including this boundary was 18 pct, whereas the total deformation of the specimen was only 7.7 pct. The sharpest ridges or valleys could be revealed by electroetching after repolishing, as shown in Figs. 9b and 10b, although reproduction of the micrographs may possibly obscure the visibility of the traces. The ridges or valleys of Figs. 9a and 10a correspond completely with the faint lines of Figs. 9b and 10b.

It is interesting to note from Fig. 10a that the effect of grain boundary sliding is by no means restricted to a thin layer of several interatomic distances thick (the thickness of grain boundary as proposed by Ke on the basis of internal friction measurement[1]), but extends to a rather large volume along both sides of the heavily slid grain boundary, in this case the affected area is about 0.2 mm wide to each side of the slid boundary.

It has been shown that the effect of etching on a grain boundary depended on the orientation difference of two neighboring grains.[2] This fact suggests that the orientation difference across the sharp

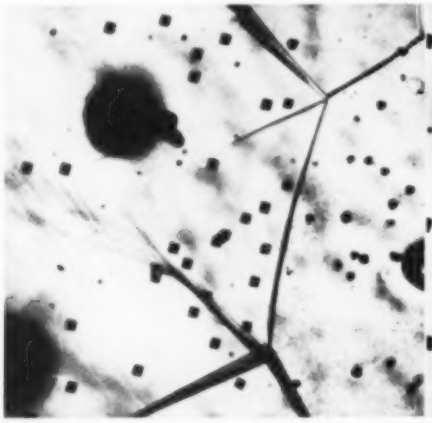


Fig. 7a—Specimen P-3. Fold formation in the left center grain caused by sliding of the opposite grain boundary. X100

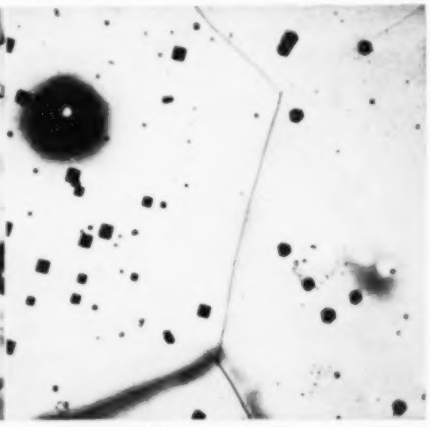


Fig. 7b—Specimen P-3. Same field as Fig. 7a repolished and etched. Folds cannot be revealed by repolishing and etching. X100

ridges or valleys of Figs. 9b and 10b were quite small but large enough to give electroetching effects. This etching effect persisted even after a surface layer of 0.02 mm thick was removed by electropolishing. Accordingly, this effect was not a surface phenomenon. That the orientation difference across this kind of boundary is quite small is further proved by the fact that the slip lines produced by a small amount of deformation at room temperature can go through this kind of boundary. This type of boundary may properly be called a "subgrain boundary," and the grains bound by them "subgrains."

It is interesting to note in Fig. 10b that the original straight grain boundary becomes angular after the process of boundary sliding and migration. In fact, every sharply bent segment along this new wavy grain boundary is due to the formation of a subgrain along this boundary. This kind of angular boundary is shown in Fig. 11a and was obtained by repolishing and etching a heavily slid grain boundary. The sketch, Fig. 11b, has been included to show the structure more clearly because of the difficulty of reproducing the fine traces. In comparison with Fig. 10a, Fig. 10b further illustrates that boundary migration is not a homogeneous process. It should be emphasized that whenever a migrating boundary encounters a scratch, etch pit, or needle mark, it may become angular; however, it may also become sharply irregular even if there are no such disturbing physical effects in its way. This phenomenon occurs more frequently and prominently in the case of heavily slid boundaries. The effect could also be caused by inclusions or other defects.

Discussion of Results

Under the conditions of stress and temperature to which these high purity aluminum specimens were subjected in creep, grain boundary sliding and migration and the resultant deformation in the grains (initiated by grain boundary sliding) are the salient features of creep. It is generally agreed that the grain boundary slides during creep;[10-13] however, grain boundary migration as a phenomenon during creep has been observed only very recently.[12-13] While the factors which govern and influence the amount, direction, and position of grain boundary sliding and migration remain to be investigated, it

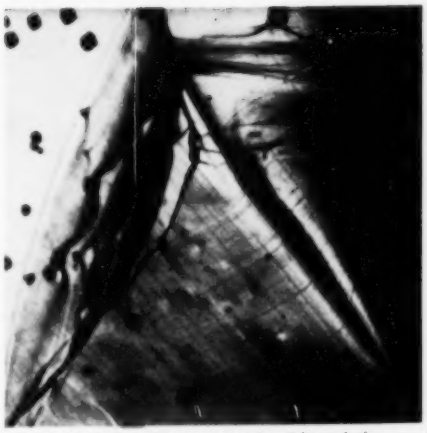


Fig. 8—Specimen P-5. Fold produced by sliding of the opposite grain boundary causing pre existing slip lines to be rather sharply bent through this fold. X100.

is definitely established from the results presented above that surface irregularities exert interesting effects on grain boundary sliding and migration and that these effects penetrate quite deeply into the thickness of the specimen.

It was observed, in some cases, that the part of the migrating grain boundary near an etch pit moves ahead of the rest of the grain boundary at the fore-side of the etch pits. When the balance of the grain boundary catches up with that portion at the etch pit and then proceeds to move on, the grain boundary at the etch pit lags behind in the migration process and appears reluctant to leave the etch pit vicinity (see Figs. 2a, 3, 6, and 8). This would suggest that the grain boundary seeks a lower surface energy position; in this case, one is offered by the etch pit.

From the results given above about grain boundary sliding and migration, it is realized that this process is a highly cooperative one, especially when several triple points are involved across the cross-section of the specimen. It was observed in general

Fig. 9a—Specimen P-3. Subgrain formation by bending around the triple point in the lower left grain. X100.

Fig. 9b—Specimen P-3. Same field as Fig. 9a repolished and etched. Subgrain boundaries only very faintly revealed around the triple point in the lower left grain. X100.

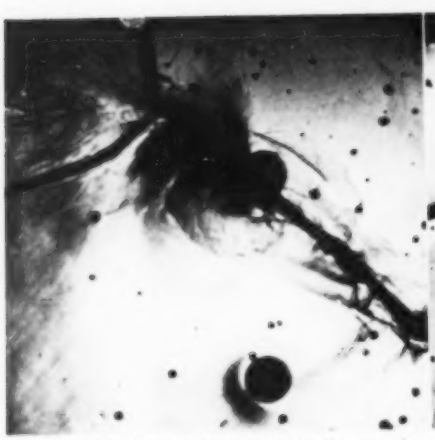


Fig. 10a—Specimen P-8. Subgrain formation by bending around the triple point and along the heavily slid grain boundary. X50.

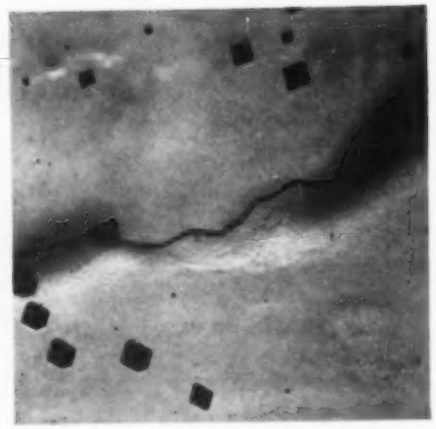
Fig. 10b—Specimen P-8. Same field as Fig. 10a repolished and etched. Final grain boundary becomes angular, subgrains revealed by electroetching after repolishing. X50.

that the grain boundary situated in a favorable position with respect to the applied stress for sliding, slides first.

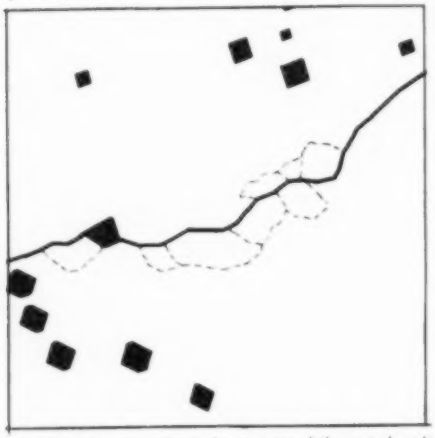
Boundary sliding will inevitably upset the original equilibrium arrangement of atoms on both sides of the slid boundary and particularly around a triple point of three grain boundaries. In other words, a high energy region has been created by boundary slip, which may either appear as stored elastic energy or may generate new surfaces through subgrain formation. The grain boundary cannot continue to slide as a result of these disturbing effects and boundary migration sets in, in order to decrease the overall energy. During the process of grain boundary migration, small deformation may take place by readjustment of atoms. When the grain boundary migrates to a more stable position, grain boundary sliding may set in again. Boundary migration is carried out by diffusion and therefore is a time-dependent process. The appearance of a creep curve obtained between two reference marks including a boundary which has undergone successive sliding and migration shows just what may be expected, namely, a multiple process of an "active boundary sliding period" and "boundary migration period."

Though the initial grain boundaries as observed at a magnification of 500X appear to be fairly straight and smooth, it is not expected that the grain boundaries could be so regarded on an atomic scale. It is reasonable to assume that the strains created during grain boundary sliding would not be uniformly distributed along both sides of the grain boundary (see Fig. 10a). The extent of nonuniform distribution of stress will be even more severe around the triple point where the sliding grain boundaries meet because of the interlocking effect of the grains. Bending in the grains would be expected to take place as a result of this nonuniform grain boundary sliding.

The subgrain boundaries are therefore believed to be formed by lattice bending as suggested by Crussard.[7] Bending is also considered to play an important role in the formation of folds or bands of



a—Micrograph at X150 showing angular nature of boundary and subgrain structure.



b—Sketch to show more clearly the structure of the grain boundary and subgrains.

Fig. 11—Specimen P-3. Appearance of boundary, originally straight, after creep test.

the type shown in Fig. 8. It has been shown that a subgrain boundary can be revealed by electroetching after repolishing, while a fold cannot. This fact suggests that the orientation difference across a subgrain boundary is rather more abrupt and appreciably larger than the orientation difference across a fold. During microscopic observations of creep, both the slid grain boundary and subgrain boundaries formed by bending did not appear angular, as shown in a repolished surface. This fact suggests that the angular nature of the final grain boundary is developed first by bending and then by a mechanism of polygonization as observed after cold bending followed by high temperature annealing.[4] Since every segment of the slid boundary is also a boundary of a subgrain and since two legs of the subgrain boundary usually rest on the slid boundary, the angularity of the slid boundaries is believed to be the result of lattice bending and subsequent subgrain formation (see Fig. 11b).

The observation of the angular nature of the boundary after a period of sliding and migration, the latter characterized by a relatively small creep strain, may help to explain the behavior of the grain boundary of the bi-crystal of tin during creep observed by King, Cahn, and Chalmers.[5] They found that the initial creep rate was rather high and then decreased with time. This behavior probably coincides with only one cycle (one period of active boundary sliding followed by a period of boundary migration, as shown in Fig. 4) of an extended creep curve measured across a grain boundary. Chalmers et al. measured lateral displacement along the grain boundary, whereas axial strain was measured in the present tests.

Since the manuscript for this report was first submitted for publication, two papers[6,7] were published in this field describing work on other materials. They show that there is a type of deformation in the grains associated with grain boundary sliding, referred to as "folds" in the present paper. Betteridge and Franklin's explanation[8] of the formation of "folds" agrees with what has been presented above; however, they did not pursue the program to examine changes occurring in the grain boundary region after extensive grain boundary sliding had taken place.

It is difficult to determine on the basis of their published micrographs whether the process of grain boundary sliding and migration has taken place in a step-wise manner. This could be explained by: 1—the use of different material, and 2—the conditions of testing such as temperature, stress, and grain size, but particularly temperature.

Summary and Conclusions

The following conclusions can be drawn from constant load creep experiments on very coarse grained, high purity aluminum at 700°F under a stress of 85 psi:

1—Successively alternating boundary sliding and migration and the resultant deformation in the grains caused by the above process are the salient features of creep at elevated temperatures.

2—A creep curve obtained between two reference marks across a grain boundary which undergoes successive sliding and migration shows several cycles of "active boundary sliding" and "boundary migration." This behavior is supported by optical observations during the actual creep test.

3—Etch pits, scratches, and needle indentations on the grain surface exert a strong influence on the course of boundary migration, mainly, as attracting effect. Surface energy around the irregularity is suggested to be a very important factor in this phenomenon.

4—The effect of grain boundary sliding is by no means restricted to a thin layer in the vicinity of grain boundary, but rather extends to a large volume along both sides of the heavily slid boundary.

5—Two types of deformation in the grains caused by boundary sliding were observed; i.e., "fold" formation and "subgrains." Lattice bending is believed to play an important role in both forms of deformation.

Acknowledgment

The authors are grateful to the Bureau of Ships for support of this research program and the permission to publish the results. Appreciation is expressed to the Aluminum Co. of America for supplying the material, and also to A. G. Bucklin and E. W. LaRocca for their help in building the equipment for this investigation.

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Technical Note

Melting Point and Transformation of Pure Chromium

by D. S. Bloom, J. W. Putman, and N. J. Grant

SEVERAL recent determinations of the melting point of pure chromium have been reported which give values of 1845°C,[1] 1892°C,[2] 1930°C,[3] 1860°C,[4] and 1890°C.[5] Because of this wide spread of values, it appeared desirable to make one additional attempt to obtain a more accurate, reliable figure. Bloom and Grant[3] recently reported a phase transformation in chromium; it was desired to check this, too, in the course of checking the melting point.

To accomplish this, a much higher purity chromium was required. Electrolytic chromium containing about 0.5 pct O was crushed to a finer size and was then annealed in highly purified, dried hydrogen at 1375°C for 100 hr. This treatment produced chromium with about 0.008 pct O, 0.002 pct N, and negligible carbon. The balance of the impurities was 0.3 pct Fe, 0.03 pct Si, 0.004 pct S, and less than 0.001 pct Mo. This chromium was melted in a stabilized zirconia crucible under prepurified argon, using induction heating. Temperature measurements were made with annealed wolfram-molybdenum thermocouples and a Leeds and Northrup Speedomax Recording Potentiometer. The thermocouples were annealed for 1 min in hydrogen at a temperature of about 2400°C.

Each leg of the thermocouple was immersed in an ice-water mixture using mechanical connectors to the lead wires. The thermocouple tip was immersed in the molten chromium in a zirconia protection tube which introduces an error of about 3°C as a temperature drop through the walls of the tube. The weight of chromium was about 183 g. The dimensions of the melt in the crucible were about 1.25 in. diam x 2 in. high. The tip of the thermocouple was held about ½ in. from the bottom of the crucible. Two new thermocouples were used with the above set-up giving the results shown in Table I.

Since this chromium is purer than that used by Greenaway, Johnstone, and McQuillan,[1] by Carlile et al.,[2] and other investigators,[4,5] it is believed to be the more accurate. Thermocouple aging effects which are believed responsible for the high value of 1930°C[3] were avoided in this work. This value of 1903°C is correct to about ±10°C.

The right hand column lists the temperature of the $\alpha \rightarrow \beta$ transformation on heating and the $\beta \rightarrow \alpha$ on cooling.[3] A sample curve is shown in Fig. 1. The value so determined is 1840°C ± 15°C. Fig. 1 gives adequate evidence that there is a transformation in chromium at high temperatures.[3]

Table I. Melting and Transformation Temperature Data

Thermocouple	Melting Point		$\alpha \rightleftharpoons \beta$ (b.c.c.) Transformation	
	Emf (mv)	Temperature, °C	Emf (mv)	Temperature, °C
No. 1	4.65 (c)*	1886		
	4.77 (h)†	1903	4.38	1848
	4.70 (c)	1894	4.15	1815
	4.80 (h)	1908	4.30	1836
No. 2	4.77 (c)	1903		
	4.83 (h)	1912		
	4.80 (c)	1908	4.43	1855
	4.82 (h)	1911	4.38	1848
Average	4.77	1903	4.33	1840

* (c) = cooling
† (h) = heating

Fig. 1—Cooling curve of chromium indicating solidification at 4.80 mv (1908°C) and transformation at about 4.43 mv (1855°C). Note deletion of 4 min of the curve during solidification period.

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Partial Titanium-Vanadium Phase Diagram

by Paul Pietrokowsky and Pol Duwez

Titanium and vanadium form a complete series of solid solutions at temperatures above 885°C. Below 885°C, vanadium is slightly soluble in α titanium (about 1.5 pct V at 650°C) and a two-phase α plus β region extends to 18.5 pct V at 650°C. The alloys up to 15.0 pct V transform into a supersaturated α solid solution upon rapid cooling from the β phase. Above 15 pct V the β phase may be retained. The temperature at which the β to α' martensite-type transformation takes place decreases steadily from 850°C for 0.5 pct V to 325°C for 12.7 pct V.

IN the transition series of the periodic table, the six elements that have a body-centered cubic structure at all temperatures, namely chromium, vanadium, molybdenum, columbium, tungsten, and tantalum, fulfill the conditions required for the existence of complete series of solid solutions with the high temperature (β) form of titanium. Up to the present time, complete solubility at temperatures above the α to β transformation of titanium has been observed in the systems Ti-Mo,[1] Ti-Cb,[2] Ti-W,[3] and Ti-Ta.[4] The two remaining elements, vanadium and chromium, have atomic diameters which are not as near that of titanium as the atomic diameters of the four other elements. Both are smaller than titanium and the difference, expressed in percentage of the titanium atomic diameter, is 12.3 pct for chromium and 8.2 pct for vanadium. On this basis, the least favorable conditions for solid-solution formation are therefore encountered in the Ti-Cr system. The early work on the Ti-Cr system[5] revealed that up to about 1100°C the solubility of chromium in β titanium is limited, and an intermetallic phase exists near the stoichiometric composition $TiCr_2$. It was found later[6] that a continuous series of solid solutions extends across the diagram at higher temperatures, and $TiCr_2$ forms by solid state reaction. It became apparent therefore that complete solubility would also be present in the Ti-V system. The present investigation confirms this possibility.

The iodide-refined titanium used in this investigation was purchased from the New Jersey Zinc Co., New York. According to the manufacturer, a typical analysis of this product is 0.0065 pct Mn, 0.0022 pct Fe, 0.0015 pct Cu, and 0.0042 pct Pb. The Vickers hardness number (10 kg load) of the crystalline bar varies between 55 and 80. The vanadium metal was purchased from the Westinghouse Electric Corp., Lamp Div., Bloomfield, N. J. The purity of this metal is probably above 99 pct V. The oxygen content must have been quite low, since a bar about $\frac{1}{4}$ in. square could be cold rolled into a ribbon 0.020 in. thick.

To prepare the various alloys on the titanium-rich side of the diagram, the titanium rod was machined into cups 1 cm in diam and about 1.5 cm high. Pieces of vanadium, of the proper weight, were cut from the square bars and placed within the cups prior to melting. On the vanadium-rich end of the diagram, single pieces of the metals, in the proper weight pro-

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Fig. 1—Lattice parameter of the β phase in Ti-V alloys quenched from 650° C.

portions, were used to make the alloys. Melting was accomplished by placing the two metals on the water-cooled hearth of a high temperature electric arc furnace and striking an arc in the presence of an atmosphere of high purity helium. The operation of such a furnace has been described in the literature.[7] After melting, the alloyed pellets were sealed in quartz tubing which had been evacuated, and homogenized at 980° C for 10 days. A few preliminary experiments indicated that this procedure was sufficient to remove coring. The homogenizing treatment was terminated by a rapid water quench. The alloys were not chemically analyzed because the change in weight after melting was generally less than 0.2 pct.

The homogenized ingots, quenched from 980° C, were quartered and the pieces sealed in quartz or Vycor tubing, depending on the temperature of the subsequent isothermal heat treatment. The following heat treating schedule was followed: 1—870° C for 10 days, 2—815° C for 10 days, 3—760° C for 10 days, 4—710° C for 20 days, and 5—650° C for 30 days. All specimens were water quenched upon completion of the heating cycle. Heat-treated samples were prepared for metallographic examination by conventional methods. The etchant was a solution of one part 48 pct hydrofluoric acid, one part concentrated nitric acid, and two parts glycerine. Powder diffraction photographs were made with a 14.32 cm Debye-Scherrer camera which utilized the Straumanis-type film arrangement. Filtered copper radiation was used throughout the entire investigation. X-ray specimens were obtained by filing the various alloys after they had been subjected to the isothermal heat treatments. The filings were sealed in quartz ampules and quenched in liquid argon from the temperature at which the corresponding solid sample had been heat treated.

Results

The results of this investigation leading to the establishment of the constitution diagram between 980° and 650° C shall be presented as follows: the existence of a continuous series of solid solutions based on the application of Vegard's law, the determination of the phase boundaries of the α, α plus β, and β regions by metallography, and the determination of the β to α'' transformation by measurement of the heat effect due to this transformation during rapid cooling from the β field.

X-Ray Diffraction Measurements. An initial X-ray investigation of the variation in lattice parameter of alloys containing from 5 to 95 pct V and quenched from 980° C established the existence of a continuous series of solid solutions. These data alone do not exclude the possibility that at lower temperatures the solid solution of β titanium and vanadium decomposes into either two phases of crystallographically similar lattice but of different compositions (as occurs in the systems Au-Ni and Al-Zn) or into intermetallic compounds (as in the Ti-Cr system). For this reason a lattice parameter vs. composition study was made at 650° C. At this temperature, alloys containing from 25 to 95 pct V were solid solutions and in this concentration range the lattice parameter varies almost linearly. However, as shown in Fig. 1, the deviation from Vegard's law is quite large for high vanadium content. The X-ray diffraction patterns of alloys containing 5, 10, and 15 atomic pct V indicated the presence of a hexagonal phase in addition to the body-centered cubic phase. In this range, the parameter of the cubic phase remains constant. The intersection of the two lines shown in Fig. 1 locates the limit of the one-phase β solid solution field at 650° C, namely 17.5 atomic pct (18.5 wt pct) V. This phase boundary was checked by the microscopic study (see below).

It is interesting to note that by extrapolation the lattice parameter of the β phase vs. concentration curve to 0 pct V leads to the fact that the lattice parameter of β titanium at room temperature would be about 3.28 kX. The difference between this parameter and that measured by Burgers at about 900° C, namely 3.32 kX, corresponds approximately to the change in volume due to thermal expansion.

Metallography. The results of the metallographic study of the Ti-V alloys are shown schematically in Fig. 2. When quenched from above the transformation temperature of pure titanium, the characteristic Widmanstätten pattern of transformed β is visible until a critical concentration of vanadium is reached and the allotropic transformation, β to α', is prevented. Fig. 3a shows a 15 pct V alloy that was quenched from 980° C into water. The microstructure indicates a stabilized β structure having equiaxed grains. The 14.8 pct V alloy, Fig. 3b, is predominately β; however, some of the decomposition product, α', is present after quenching from 980° C. The

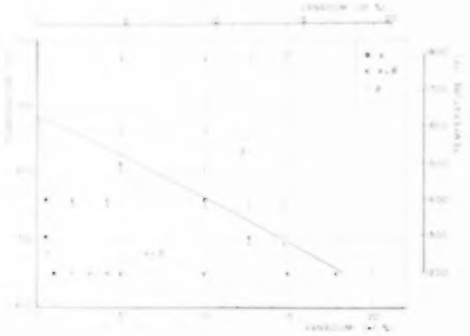
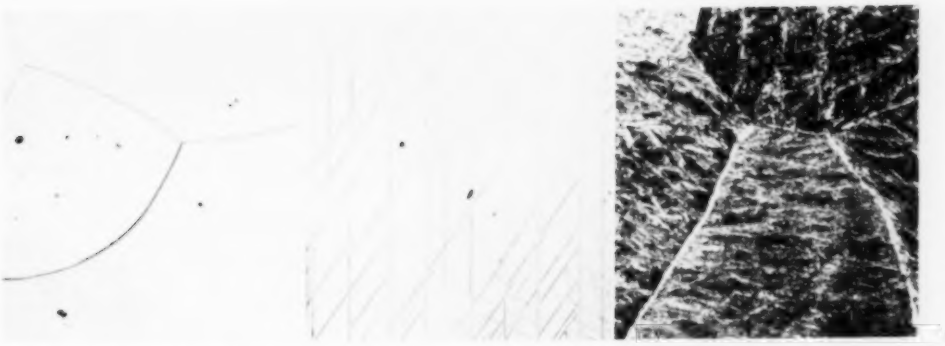


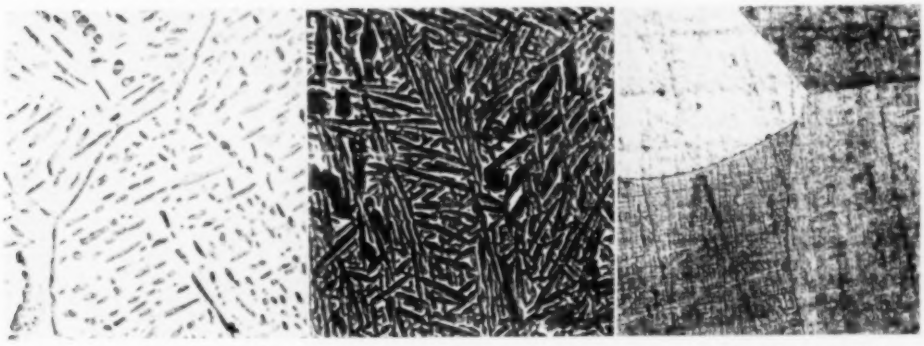
Fig. 2—Partial phase diagram of the Ti-V system. Symbols with vertical arrows correspond to specimens in which β transformed to α'' during quenching.



a—15 pct V quenched from 980°C (β retained).

b—14.8 pct V quenched from 980°C (mostly β plus α″ needles).

c—10 pct V quenched from 760°C (α and α′).



d—10 pct V quenched from 650°C (α and β).

e—5 pct V quenched from 650°C (α and β).

f—0.5 pct V quenched from 760°C (α).

Fig. 3—Typical microstructure of various Ti-V alloys.

minimum concentration of vanadium necessary to stabilize the β structure is therefore approximately 14.9 pct. When quenched from isothermal soaking temperatures below the transformation temperature of pure titanium, the microconstituents assume characteristic appearances depending on whether or not the equilibrium β, at temperature, transformed to α″, either in part or entirety, after being quenched in water. Fig. 3c shows a 10 pct V alloy that was water quenched after receiving a thermal treatment at 760°C. Here the equilibrium β has decomposed upon quenching. The same alloy, quenched from 650°C, is shown in Fig. 3d. At 650°C, the vanadium content of the equilibrium β is greater than the critical composition necessary to retain β. A 5 pct V alloy heat treated at 650°C is shown in Fig. 3e. As in Fig. 3d, the dark etching α titanium is in equilibrium with the light etching β.

On the basis of the microstructure of the alloys corresponding to the symbols shown in Fig. 2, the boundary between the α plus β and the β fields was traced as shown in the same figure. This boundary passes through a point located at 18.5 pct V at 650°C, as required by the X-ray diffraction study (see above).

Although no extensive study was made in the range of 0 to 2 pct V, the limit of solubility of vanadium in α titanium was located approximately. The microstructure of alloys containing 0.5 and 1 pct V indicated that these alloys were α solid solution at 650°C and the 0.5 pct V alloy was also α solid solution after the 760°C heat treatment (Fig. 3f). Since, on the other hand, the alloy containing 2 pct V was in the α plus β field at 650°C, the boundary between α and α plus β was traced tentatively as indicated on Fig. 2.

β to α″ Transformation: Because the alloys containing less than 14.9 pct V transform from β to a supersaturated α solid solution (α′) during quenching, it was anticipated that the temperature at which this transformation takes place could be detected by thermal analysis.

The method for measuring transformation temperature at very fast cooling rates consists essentially of heating a small sample of metal to which thermocouples are spot welded, cooling the sample by means of a jet of helium, and recording the temperature vs. time curve on an oscillograph. This method, used by Greninger in his study of the martensite arrest in carbon steels,[7] has been more recently applied to titanium alloys, and a description of the experimental apparatus has been given.[8]

Alloys containing up to 14.8 pct V were tested and thermal arrests were recorded at various rates of cooling in the range of 180° to 9000°C per sec. For the alloys containing up to 12.7 pct V, a clearly defined thermal arrest was observed. Within the limits of experimental error (estimated to be ±4°C), the

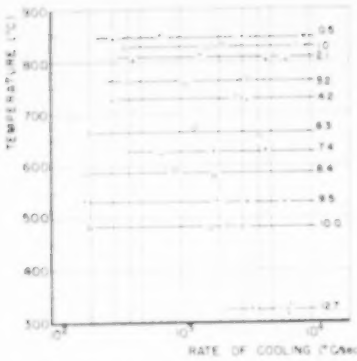


Fig. 4—Effect of rate of cooling on the transformation temperature of Ti-V β solid solution for various vanadium concentrations.

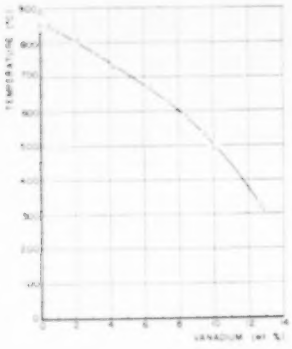


Fig. 5—Effect of vanadium content on the transformation temperature of rapidly cooled Ti-V alloys.

rate of cooling had no effect on the temperature at which the thermal arrest occurred, Fig. 4. As the vanadium content increases, the transformation temperature decreases progressively, as shown in Fig. 5.

Thermal analysis experiments made on alloys containing 14.8 and 15 pct V failed to reveal a thermal arrest. This is not surprising for the 15 pct V, since the structure of this alloy was retained β when quenched from high temperature. For the 14.8 pct V alloy, the microstructure as shown in Fig. 3b indicated that some transformation took place during quenching. The failure to detect a thermal arrest for this alloy is due to the lack of sensitivity of the thermal analysis method, when only a small portion of the specimen undergoes transformation. It is therefore logical to assume that, if the transformation temperature could be measured by a more sensitive method, the curve of Fig. 4 would extend to lower temperature and cross the composition axis at about 14.9 pct V.

An X-ray diffraction study of the alloys quenched from 980°C indicated that both the β and α' structures were present in the alloys containing from about 4 to 12.7 pct V. Judging from the relative intensity of the α and the β reflections in the patterns, it was quite clear that the amount of the retained β phase was increasing with vanadium content. Similar results had been found previously in Ti-Mo alloys.[7]

As shown in a previous paper,[7] the β to α transformation in pure titanium is progressively lowered when the rate of cooling increases from a few degrees per second to about 10,000°C per sec. This behavior is indicated on the vertical axis of the graph of Fig. 5 by two points limiting a temperature range from 885° to 855°C. An extrapolation of the curve of transformation temperature vs. vanadium content to 0 pct V seems to pass through a temperature corresponding to that of pure titanium obtained under very high cooling rates (about 855°C). This result suggests that the transformation in the alloys of low vanadium content might also be affected by the rate of cooling in the range which was not covered in the present study, namely from about 5° to 180°C per sec. At relatively slow cooling rates, however, the equilibrium reaction β to α plus β could take place, at least partially, and in such a case, the thermal analysis measurement should be supplemented by microscopic observations. Obviously more detailed studies of the transformation kinetics should be carried out in the range of cooling rates from a few degrees per minute to about 100°C per sec.

Conclusions

1—Titanium and vanadium are completely soluble in each other above the transformation temperature of titanium. The solubility of vanadium in α titanium is between 1 and 2 pct at 650°C and the α plus β field extends to 18.5 pct at 650°C.

2—Alloys containing up to 14.8 pct V undergo a martensite-type reaction from β solid solution to a supersaturated α solid solution. The temperature at which this reaction takes place is independent of the rate of cooling, within the range from 180° to 9000°C per sec, and decreases with vanadium content. Above 15 pct V, the β structure can be retained by quenching.

Acknowledgment

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Crystal Structure of ZrB_{12}

by Benjamin Post and Frank W. Glaser

ALTHOUGH most transition metals form a wide variety of boride compounds, the existence of only one zirconium boride, ZrB_2, had been established prior to this investigation.[1] The crystal structure and some properties of a hitherto unreported zirconium boride, ZrB_{12}, are described in this paper. Only one other dodecaboride of a transition metal, UB_{12}, has been reported up to the present.[2]

Chemical analysis of the new compound showed 44 pct Zr and 56 pct B, compared with 41 pct Zr and 59 pct B computed for ZrB_{12}. The difference is within the range of error of the analytical method employed.

ZrB_{12} shows marked metallic properties. It is obtained as a fine black powder. Measurements of electrical resistivity were made at −79°C, +22°C and +64°C. At 22°C the electrical resistivity is approximately 60 microhm-cm. Over this range the temperature coefficient of resistivity is +0.00162. The thermal conductivity at room temperature is 0.122 watts per cm per °C. The product of the electrical resistivity and thermal conductivity is therefore 8.05, which is not far from the normal Wiedemann-Franz ratio (approximately 7.5 at room temperature). These measurements were made on hot pressed samples which were not always high density specimens. The hardness of ZrB_{12} on the Rockwell A scale varies between 92 and 92.5.

Crystal Structure Determination

Single crystals of ZrB_{12} of a size suitable for X-ray diffraction analysis could not be obtained and powder samples had to be used. Filtered Cu radiation was used throughout.

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Powder diagrams were indexed on the basis of a face-centered cubic unit cell. After correction for film shrinkage, a Bradley and Jay extrapolation[3] to $\theta = 90°$ indicated that $a_0 = 7.408 \pm 0.002$A.

The volume of the unit cell is 406.5A.[3] The assumption that there are four "molecules" of ZrB_{12} per unit cell leads to an X-ray density of 3.63 g per cc. The measured density is 3.7 g per cc (as measured by the immersion method).

Relative intensities of reflections were determined by integrating the intensities of diffraction peaks using the count register of a North American Philips wide range spectrometer. Intensities measured in this way showed good agreement with photographic intensities estimated visually using multiple film techniques.

The usual Lorentz, polarization, and multiplicity corrections were applied to the observed intensity measurements.

There are striking similarities between ZrB_{12} and UB_{12}. The unit cell of the latter is also face-centered cubic and contains four molecules. $a_0 = 7.473$, compared with 7.408 for ZrB_{12}. The radius ratio rB/rU is 0.57; rB/rZr is 0.54.

In both ZrB_{12} and UB_{12} the four metal atoms in each unit cell are in special positions; only the boron positions need be determined. The latter may be inferred from the extent to which diffraction of X-rays by the 48 boron atoms reinforces or weakens the diffraction by the metal atoms. This effect is relatively much greater in the case of the zirconium compound.

In their determination of the crystal structure of UB_{12}, Berthaut and Blum[2] assumed holohedral symmetry (space group O_h^5, $Fm3m$) and reported that the U atoms are in positions 4(a): 000; $0\frac{1}{2}\frac{1}{2}$; $\frac{1}{2}0\frac{1}{2}$; $\frac{1}{2}\frac{1}{2}0$; the boron atoms were reported to be in positions 48(i) with $x = 1/6$: $\frac{1}{2}xx$; $\frac{1}{2}\bar{x}x$; $x\frac{1}{2}x$; $\bar{x}\frac{1}{2}x$; $xx\frac{1}{2}$; $x\bar{x}\frac{1}{2}$; $\frac{1}{2}xx$; $\frac{1}{2}\bar{x}x$; $x\frac{1}{2}x$; $x\frac{1}{2}x$; $xx\frac{1}{2}$; $x\bar{x}\frac{1}{2}$.

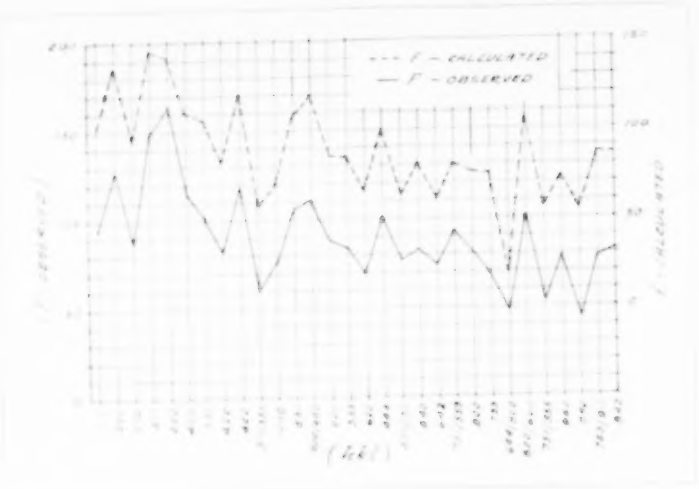

Fig. 1—Values of $F_{obs.}$ and $F_{calc.}$ for ZrB_{12}.

Holohedral symmetry was also assumed for ZrB_{12}. It was evident from the relative diffraction intensities that the value of x in positions 48(*i*) must be in the range 0.150 to 0.175. (Positions 48(*g*) may be ruled out on steric grounds since they lead to B-B distances of 1.2A compared with the usual B-B distance of 1.7A. Positions 48(*h*) are equivalent to 48(*i*), differing only in choice of origin.)

Structure factors were computed for a range of values of x from 0.125 to 0.200. Graphical comparison of these with values of $F_{obs.}$ showed best agreement at $x = 1/6$. Values of $F_{obs.}$ and $F_{calc.}$ for $x = 1/6$, are shown in Fig. 1.

Discussion

Twenty-four boron atoms are arranged, octahedrally, about each zirconium atom in ZrB_{12}. Each boron atom in turn, is equidistant from two zirconium atoms. The arrangement of boron atoms in the unit cell is best visualized by considering a regular octahedron about each zirconium atom to be cut by a plane perpendicular to the line from the central zirconium atom to the vertex of the octahedron. A boron atom is located at each of the four corners of the square formed by the intersection of one plane and octahedron; six such squares are formed about each zirconium atom. The arrangement of boron atoms about two adjacent zirconium atoms is shown in Fig. 2. All zirconium atoms in one unit cell are shown; in order to show the boron arrangement clearly, all boron atoms other than those about two adjacent zirconium atoms have been omitted from the drawing.

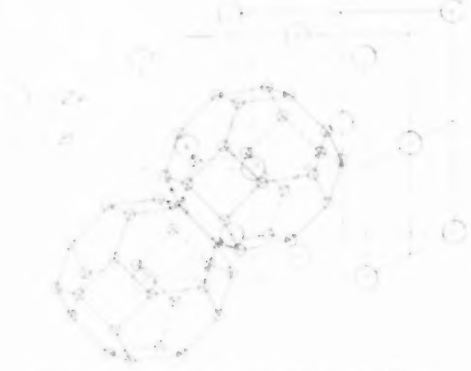

Fig. 2—Arrangement of boron atoms about two adjacent zirconium atoms in ZrB_{12}.

Each boron atom has five nearest boron neighbors at B-B = 1.75A. The five B-B bonds formed by one boron atom are indicated by arrows in Fig. 2. Each boron atom is also equidistant from two zirconium atoms at B-Zr = 2.76A. The B-B distances are normal, but the metal to boron distance is much greater than in ZrB_2 (2.54A).[1] This increase in bond length is due, in part at least, to the high coordination number of the metal atoms. Kiessling[2] has commented on the abnormally long metal to boron bonds in MB_{12} compounds where the metal coordination number is also 24. The U-B distance in UB_{12} is also much greater than "normal."[3]

Acknowledgments

The authors wish to express their appreciation to the Office of Naval Research for the support of this investigation and permission to publish the results. They also gratefully acknowledge valuable discussions with R. Steinitz of the American Electro Metal Corp.

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[2] F. Berthaut and P. Blum: *Comptes Rendus* (1949) **229**, p. 667.
[3] A. J. Bradley and A. H. Jay: *Proc. Physical Soc., London* (1932) **44**, p. 503.

Effect of Zirconium on Magnesium-Thorium And Magnesium-Thorium-Cerium Alloys

by T. E. Leontis

Data are presented in this paper to show that addition of zirconium to sand-cast Mg-Th alloys effects a marked decrease in the grain size of these alloys which is accompanied by a significant increase in the mechanical properties over the entire range of thorium content investigated. The beneficial effect of zirconium on the strength properties is maintained at elevated temperatures up to 700°F. In addition the alloys exhibit exceptionally high creep resistance at temperatures up to 600°F. Zirconium does not greatly improve the strength or creep characteristics of extruded Mg-Th alloys. Cerium is not a desirable addition to Mg-Th-Zr alloys.

IN a previous paper,[1] it was shown that addition of thorium to magnesium imparts exceptionally high resistance to creep at elevated temperatures both in the sand-cast and in the extruded states. The large grain size of the sand-cast alloys, however, renders them of questionable value as engineering materials. Sauerwald[2] showed that the grain size can be reduced effectively by the addition of zirconium. Results of a detailed study of the properties of Mg-Th-Zr alloys over a wide range of thorium content are presented in this paper. In accordance with the preliminary findings of Sauerwald,[2] it is shown here that Mg-Th-Zr alloys possess the unusual combination of fine grain size and a high level of strength and creep resistance at elevated temperatures. Some information on the effect of cerium on the properties of Mg-Th-Zr alloys is presented also.

Effect of Th on Solubility of Zr in Molten Mg

Sand-cast test bars and 3 in. diam extrusion billets of Mg-Th-Zr and Mg-Th-Ce-Zr alloys* were prepared according to the procedures described in the previous paper.[1] The zirconium was added in the form of sponge metal as supplied by the Bureau of Mines.[3]

The maximum amount of zirconium that usually can be introduced into magnesium at the customary alloying temperature of 1350°F is about 0.7 pct. The analyses in Table I clearly show that thorium increases the solubility of zirconium in liquid magnesium. The tabulated zirconium contents are designated as "acid soluble;" this serves to distinguish these values from the small amount of zirconium that is not dissolved when the alloy is subjected to the action of a dilute acid solution. The "acid-insoluble" zirconium is believed to be ineffective as an alloying ingredient. The acid-insoluble zirconium contents ranged between 0.03 and 0.16 pct in the present alloys, the tendency was toward higher values in alloys with higher thorium content.

In contrast to the effect of thorium, cerium decreases the solubility of zirconium in magnesium. This is shown by the three Mg-Th-Ce-Zr alloys† included in Table I and stands in agreement with the observations of Nelson and Strieter[4] on Mg-mischmetal-Zr alloys.

Effect of Zr on Grain Size of Mg-Th Alloys

The potent effect of zirconium as a grain refiner has been demonstrated for many types of magnesium alloys.[5-9] Of greatest importance to date are Mg-Zn-Zr alloys[6,7,9] and Mg-mischmetal-Zr alloys.[5,7,9] Zirconium has also been found to exert a marked grain-refining action on Mg-Th alloys. All the Mg-Th-Zr alloys cast in the form of ½ in. diam test bars have an exceedingly fine grain size (Table I). Reference to the previous paper[1] will show that binary Mg-Th alloys containing up to 6 pct Th are almost completely columnar even in the ½ in. diam reduced section of a test bar, and that 10 to 50 pct Th alloys have an equiaxed grain structure of 0.03 to 0.08 in. The data in Table I also show that addition of cerium to Mg-Th-Zr alloys has no effect on the grain size. In addition to increasing the strength properties, the fine grain size of Mg-Th-Zr alloys

* The thorium metal used in making these alloys was furnished by the U. S. Atomic Energy Commission.

† The cerium used in making these alloys had a total rare-earth content of 97.1 pct, of which less than 0.3 pct was rare-earth metals other than cerium. The principal impurities making up the balance were iron (1.49 pct) and magnesium (1.24 pct).

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Table I. Analyses, Grain Size, and Heat Treatment of Mg-Th-Zr and Mg-Th-Ce-Zr Alloys

Analyses, Pct				Sand-Cast Alloys			Extruded Alloys		
				Grain Size, 0.001 In.			Grain Size, 0.001 In.		
Th	Acid Soluble Zr	Acid Insoluble Zr	Ce	As-Cast	SHT*	Solution Heat Treatment	As-Extruded	SHT*	Solution Heat Treatment
0	0.69	0.03		2-6	2-6	1050F (2 hr)	0.5-1	2-4	1050F (2 hr)
0.93	0.82	0.08		2	3-4	1050F (2 hr)	1	4	1050F (2 hr)
1.84	0.82	0.12		1-2	3-4	1050F (2 hr)	1	4	1050F (2 hr)
3.34	0.94	0.12		2	2	1050F (2 hr)	0.2	2	1050F (2 hr)
3.34	0.94	0.09		1-2	1-2	1050F (16 hr)	0.2	1-2	1050F (4 hr)
6.05	0.94	0.09		1-2	1-2	1050F (24 hr)	0.2	0.6	1050F (4 hr)
10.00	0.90	0.16		1-2	1-2	1050F (24 hr)	0.2	0.6	1050F (4 hr)
10.00	1.01	0.15		1-2	1-2	1050F (24 hr)	0.2	0.6	1050F (4 hr)
3.37	0.97	0.05	1.25	2	2-3	1050F (16 hr)	0.4	1-2	1050F (4 hr)
2.90	0.92	0.08	2.20	2	2-3	1025F (16 hr)	0.4	1-2	1025F (4 hr)
3.15	0.97	0.06	2.93	2-3	2-3	1025F (16 hr)	0.3	1-2	1025F (4 hr)

* SHT — Solution heat treated.

is a highly desirable characteristic from the standpoint of castability and general handling in foundry operations.

The grain size of extruded Mg-Th alloys is also reduced by the addition of zirconium but not as markedly as observed in the cast alloys. The values given in Table I compare with a range of 0.001 to 0.0005 in. for binary alloys in the as-extruded condition covering the same range of thorium content.

Heat Treatment

The heat-treating time and temperature for each alloy in Table I were selected on the basis of three considerations: 1—safety from melting, 2—maximum solution and coalescence of intermetallic compounds, and 3—minimum increase in grain size.

The solidus temperature of each alloy as determined by the metallographic technique is given in Table II. The same values were obtained on specimens in three different conditions: 1—as-cast, 2—cast and heat treated at 1050°F (16 hr), and 3—as-extruded. The addition of zirconium raises the eutectic temperature of Mg-Th from 1088° to 1092°F. However, there appears to be a decrease in the maximum solubility of thorium in magnesium resulting from the addition of zirconium. From the corresponding solidus measurements it was postulated that the maximum solubility of thorium in magnesium lies between 5.8 and 10.2 pct, whereas the data in Table II indicate that in the presence of zirconium the solubility lies between 1.84 and 3.34 pct. Additions of cerium effect a significant decrease in the solidus temperature. On the basis of these determinations, all alloys except those containing 1.02 and 2.20 pct Ce were heat treated at 1050°F and the latter two compositions were heat treated at 1025°F.

Grain size measurements after heat treating at the selected temperatures for a series of time intervals between 1 and 24 hr show that Mg-Th-Zr alloys containing 6 pct Th or more and all Mg-Th-Ce-Zr alloys suffer no grain growth up to 24 hr heat treatment. Alloys of lower thorium content increase in grain size with increasing time of heat treatment. However, short heat treatments are adequate for dissolving and coalescing the small amount of compound in alloys containing up to 3.34 pct Th. Thus, the time of heat treatment used for each alloy is as given in Table I.

In the case of the extruded alloys, it was found that the increase in grain size suffered by each composition is essentially independent of heat-treating time between 1 and 24 hr. Accordingly, the extruded alloys were heat treated as shown in Table I in order to achieve maximum solution and coalescence of the intermetallic compound. In general, this can be accomplished by a shorter heat treatment in extruded alloys than in cast alloys of equivalent composition.

Throughout the paper, the following designations are used to depict the various conditions of heat treatment:

T5—Direct age at 400°F (16 hr).
T4—Solution heat treated as given in Table I.
T6—T4 + age at 400°F (16 hr).

Mechanical Properties of Sand-Cast Alloys

Tensile properties of sand-cast Mg-Th-Zr alloys were determined on the previously described cast-to-shape test bars without machining the surface and according to the methods described earlier.[1,2,10] The pronounced strengthening effect of thorium over a wide temperature range is clearly shown by the curves in Figs. 1 and 2. The decrease in tensile strength at room temperature in higher thorium contents results from the increasing amount of undissolved intermetallic compound. This is also reflected by the rapidly decreasing elongation with increasing thorium content. Comparison of the data

Table II. Solidus Temperatures of Alloys

Composition, Pct Acid Soluble			Solidus
Th	Zr	Ce	Temperature, °F
0	0.69		~1178
0.93	0.82		~1178
1.84	0.82		1158
3.34	0.94		1092
6.05	0.94		1092
10.00	0.90		1092
10.20	1.01		1092
3.37	0.97	1.25	1063
2.90	0.92	2.20	1063
3.15	0.97	2.93	1077

Table III. Tensile Properties of Sand-Cast Mg-Th-Zr Alloys in the As-Cast and in the Solution Heat-Treated Conditions at Room Temperature

		As-Cast*			T4 Condition		
Th, Pct	Acid Soluble Zr, Pct	Pct E	TYS	TS	Pct E	TYS	TS
0	0.69	20.0	8.4	25.5	11.5	6.2	22.0
0.93	0.82	16.5	10.4	25.0	20.0	8.0	23.9
1.84	0.82	12.0	11.2	24.2	16.0	11.2	25.7
3.34	0.94	5.0	13.0	20.5	10.6	12.8	25.8
6.05	0.94	3.2	14.1	20.8	8.5	13.4	25.2
10.20	0.90	2.2	16.2	22.5	4.0	15.5	23.2
10.70	1.01			19.8	0.3	17.7	19.4

*Pct E — Percent elongation in 2 in.
TYS — Tensile yield strength in 1000 psi.
TS — Tensile strength in 1000 psi.

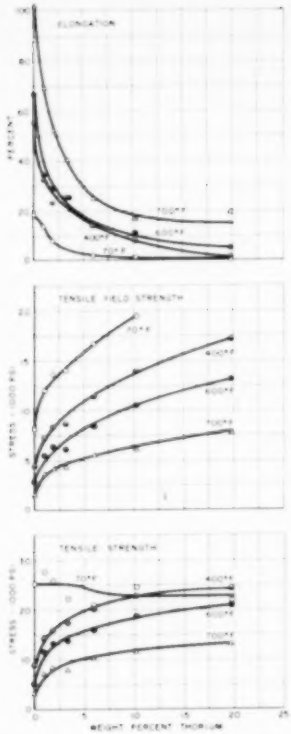


Fig. 1 — Tensile properties of sand cast Mg-Th-Zr alloys. T5 condition.

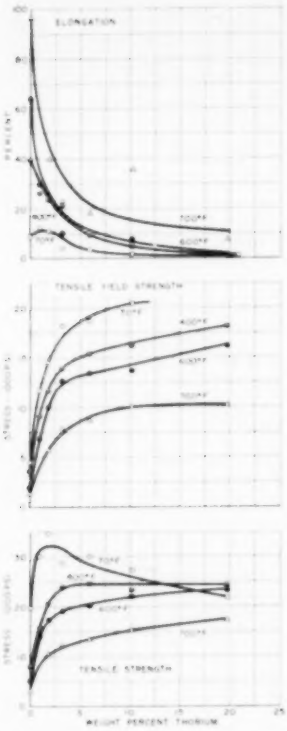


Fig. 2 — Tensile properties of sand cast Mg-Th-Zr alloys. T6 condition.

in Figs. 1 and 2 shows that the strength obtained in the T6 condition is decidedly higher than that of the same compositions in the T5 condition at all temperatures. The room temperature tensile properties for the as-cast and the T4 conditions are given in Table III. It is evident that Mg-Th-Zr alloys exhibit a much greater degree of age hardening after solution heat treatment than is obtained upon directly aging the as-cast material. In order to illustrate the beneficial effect of the addition of zirconium to Mg-Th alloys, the properties of Mg-3.34 pct Th-0.94 pct Zr alloy are compared with those of the binary Mg-3.2 pct Th alloy taken from the previous paper[1] in Table IV. The 3 pct Th composition has been selected for this comparison because it appears to be the optimum composition from all considerations. The addition of zirconium enhances the properties over the entire range of thorium content investigated; however, the increase in strength is considerably greater in alloys containing up to 6 pct Th. Another important observation to be made from the data in Table IV is that the increase in room temperature strength is realized without any sacrifice in ductility. As a matter of fact, the elongations of alloys with lower thorium contents are increased markedly by the addition of zirconium.

Table IV. Comparison of Properties of Mg-Th-Zr and Mg-Th Alloys

Composition	Properties in the T6 Condition								
	Room Temperature			400°F			600°F		
	Pct E	TYS*	TS*	Pct E	TYS*	TS*	Pct E	TYS*	TS*
3.2 Th	4.0	9.8	20.4	13.5	7.5	16.3	16.5	6.6	14.8
3.34 pct Th-0.94 pct Zr	4.0	18.2	29.1	21.3	14.0	24.2	9.7	12.8	19.2

* 1000 psi.

The elevated-temperature tensile properties of sand-cast Mg-2.5 pct Th-0.6 pct Zr in the as-cast state presented by Sauerwald[2] are somewhat lower than those obtained on Mg-1.84 pct Th-0.82 pct Zr in the T5 condition in the present investigation. This difference may be a result of the lower zirconium content of Sauerwald's alloy. On the other hand, the difference may be a result of the aging treatment used in the present work which has been found to increase the room temperature strength of these compositions by the same amount as is represented by the difference between Sauerwald's and our data at elevated temperatures.

Creep tests were conducted in the same manner as described earlier,[1,3,4] and the results are presented in the form of the following three parameters obtained by interpolation of log stress vs. log extension plots of the original data:

1—Creep limit based on 0.1 pct creep extension in 100 hr

2—Creep limit based on 0.2 pct total extension in 100 hr

3—Creep limit based on 0.5 pct total extension in 100 hr

The curves in Figs. 3 and 4 show the increase in creep limits of Mg-Th-Zr alloys with increasing thorium content. Significantly higher creep resist-

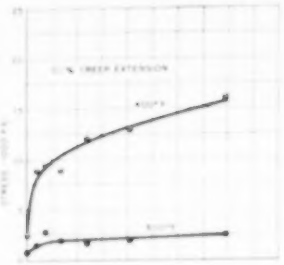


Fig. 3—Creep limits of sand-cast Mg-Th-Zr alloys. T5 condition.

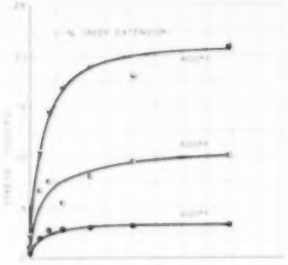


Fig. 4—Creep limits of sand-cast Mg-Th-Zr alloys. T6 condition.

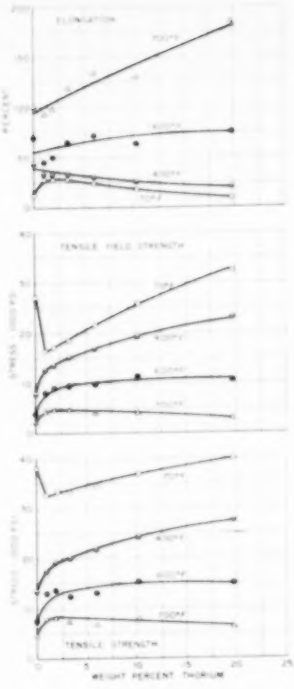


Fig. 5—Tensile properties of extruded Mg-Th-Zr alloys. T5 condition.

ance is obtained in the T6 condition than in the T5 condition. The addition of zirconium to Mg-Th alloys markedly increases the creep resistance at 400° and 500°F but has little if any effect on the creep resistance at 600°F. One of the most important observations to be made from the results presented in this paper is the high level of creep resistance of sand-cast Mg-Th-Zr alloys at temperatures up to 600°F coupled with the fact that these alloys are fine grained and can be expected to have excellent foundry characteristics. The retention of high creep resistance in Mg-Th alloys at 500° and 600°F after the addition of zirconium and the attendant grain refinement is contrary to the behavior of magnesium-mischmetal alloys upon similar treatment.[7]

The effect of additions of pure cerium on the tensile properties of Mg-3 pct Th-1 pct Zr alloy is shown by the data in Table V, where the corresponding properties of Mg-3 pct Th-1 pct Zr and Mg-6 pct Th-1 pct Zr are included for purposes of comparison.

Table V. Tensile Properties of Sand-Cast Mg-Th-Ce-Zr Alloys

			As-Cast						T5 Condition											
				Room Temperature			Room Temperature			400°F			500°F			600°F			700°F		
Th, Pct	Ce, Pct	Acid-soluble Zr, Pct	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS
3.34		0.94	3.0	11.8	20.5	3.8	13.8	22.0	25.0	8.6	17.2	20.8	8.3	15.8	25.0	6.0	13.2	39.5	4.1	8.1
3.47	1.25	0.97	7.2	15.0	20.7	2.0	17.6	23.0	13.0	12.2	22.4	15.4	11.1	21.8	36.6	8.0	18.6	64.8	5.0	9.2
2.90	2.20	0.91	1.2	14.9	16.1	0.7	17.8	21.2	5.5	13.6	21.1	8.4	11.7	21.4	26.0	8.0	17.2	66.2	5.0	9.0
3.15	2.93	0.87	0.7	16.2	18.0	0.2	18.6	20.4	1.2	13.4	19.2	9.6	11.6	21.8	30.6	8.0	16.5	86.8	4.9	8.8
6.05		0.94	3.2	14.1	20.8	1.8	16.5	21.0	14.0	11.1	20.3	13.0	10.8	18.8	14.0	8.1	15.7	24.8	5.2	10.4

			T4 Condition						T6 Condition											
			Room Temperature			Room Temperature			400°F			500°F			600°F			700°F		
			Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS
3.34		0.94	10.0	12.0	25.0	4.0	18.2	29.1	21.3	14.0	24.2	22.6	13.9	22.4	9.7	12.8	19.2	10.3	7.8	12.3
3.47	1.25	0.97	9.0	14.4	22.4	3.0	17.6	25.8	10.0	14.2	24.6	13.0	14.1	24.0	30.3	11.4	19.6	44.0	5.8	11.4
2.90	2.20	0.91	5.0	14.9	23.2	2.7	18.3	26.6	7.4	15.0	24.7	9.7	15.1	24.1	13.0	12.2	21.6	43.3	5.9	10.0
3.15	2.93	0.87	3.5	15.0	20.8	1.0	18.6	23.8	8.6	15.0	24.6	11.6	15.4	25.6	15.6	11.9	20.4	75.0	5.5	10.2
6.05		0.94	8.5	13.4	23.2	3.8	18.7	30.2	9.0	15.4	24.7	15.0	15.2	24.2	8.4	13.4	20.2	8.3	8.8	13.6

* Pct E = Percent elongation in 1.5 or 2 in.

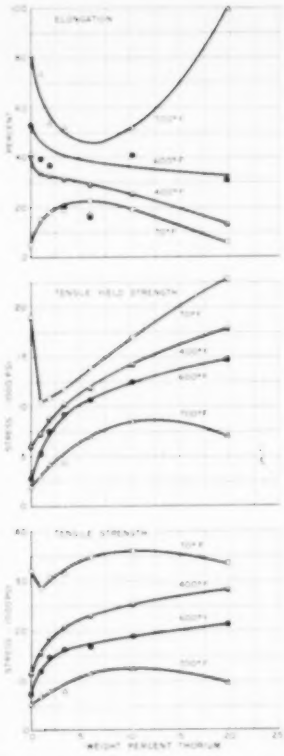


Fig. 6—Tensile properties of extruded Mg-Th-Zr alloys. T6 condition.

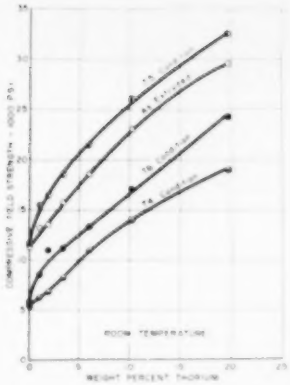


Fig. 7—Compressive yield strength of extruded Mg-Th-Zr alloys at room temperature.

The reduced section of test bars of Mg-Th-Ce-Zr alloys were machined to 0.375 in. diam and the elongation was measured over 1.5 in. gage length. Machining the surface of these alloys is necessary to eliminate the zone of grain boundary oxidation that occurs during heat treatment. Cerium effects some increase in the strength properties in the T5 condition, but the increase is accompanied by a drastic decrease in ductility, particularly at room temperature. In the T6 condition, there is no benefit realized from the addition of cerium, in fact at the higher temperatures the cerium-containing alloys have somewhat lower strength than the corresponding Mg-Th-Zr alloys.

Test bars of Mg-Th-Ce-Zr alloys for creep tests were also machined in the reduced section in order to eliminate the internally oxidized zone. For these tests, however, the bars were machined to a diameter of 0.450 in. The creep limits obtained from these tests are compared with those of Mg + 3 pct Th + 1 pct Zr and Mg-6 pct Th-1 pct Zr alloys in Table VI. In some cases where the creep limits of the Mg-Th-Zr deviate markedly from the trends established by the complete series of alloys, the values given in Table VI are those read from the curves instead of the actual experimental determinations on the particular alloy. In the T5 condition, cerium increases somewhat the creep limits of Mg-Th-Zr alloys at 400°F, particularly those based on total extension, but has no significant effect on the corresponding values at 600°F. In the T6 condition, the creep resistance of Mg-Th-Zr alloys is definitely impaired by the addition of cerium. At 400°F, this is manifested only in the creep limits based on creep extension, which, incidently, are the quantities most closely related to creep rate. In general, the results presented here show that cerium is not a desirable addition to Mg-Th-Zr alloys.

Mechanical Properties of Extruded Alloys

Properties of the alloys in the extruded state were determined on ½ in. diam rods extruded from 3 in.

Table VI. Creep Limits of Sand-Cast Mg-Th-Ce-Zr Alloys

			100-Hr Creep Limits (1000 psi)								
			400°F			500°F			600°F		
			Extension			Extension			Extension		
Th Pct	Ce Pct	Acid Soluble Zr, Pct	0.1 Pct Creep	0.2 Pct Total	0.5 Pct Total	0.1 Pct Creep	0.2 Pct Total	0.5 Pct Total	0.1 Pct Creep	0.2 Pct Total	0.5 Pct Total
T5 Condition											
3.34		0.94	10.2*	7.7*	10.0*				1.9	2.0	2.5
3.37	1.25	0.97	11.5	8.5	12.4	4.5	4.6	6.9	1.6	1.9	2.7
2.90	2.20	0.91	12.2	9.0	12.5	4.8	4.9	6.3	1.6	1.8	2.2
3.15	2.93	0.87	12.8	11.0	13.2	4.5	4.7	7.3	1.4	1.7	2.2
6.05		0.94	12.1	9.6	11.8				1.6	2.0	2.4
T6 Condition											
3.34		0.94	17.0	11.2	14.7	7.5*	6.5*	9.5*	2.9	3.1	3.6
3.37	1.25	0.97	14.0	13.0	15.0	6.2	6.1	7.8	1.8	2.1	2.7
2.90	2.20	0.91	15.0	12.0	15.0	5.7	5.7	8.0	1.7	2.0	2.5
3.15	2.93	0.87	14.5	11.0	14.3	5.0	5.2	7.2	1.9	2.1	2.8
6.05		0.94	19.0	11.1	15.5	8.0	7.1	10.1	2.0	3.3	4.3

* These values were read from creep limit vs. pct Th curves in Figs. 3 and 4.

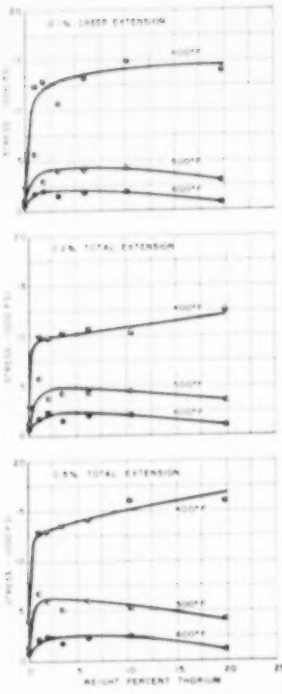


Fig. 8—Creep limits of extruded Mg-Th-Zr alloys. T5 condition.

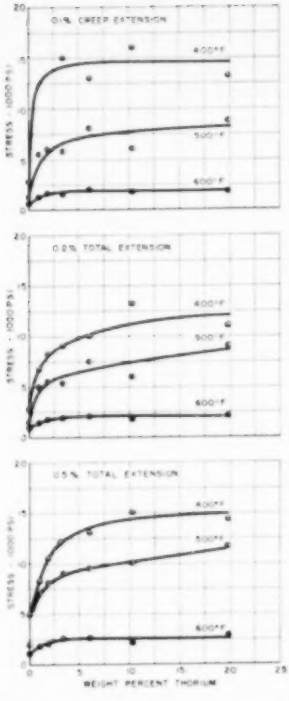


Fig. 9—Creep limits of extruded Mg-Th-Zr alloys. T6 condition.

diam billets scalped to a diameter of 2 15/16 in. and a length of 9¼ in. as limited by the size of the container on a 500 ton Wattson-Stillman direct-extrusion press. The conditions of extrusion were as follows:

Billet preheat	—	925° F (2 to 16 hr)
Container temperature	—	900° F
Die temperature	—	900° F
Extrusion speed	—	10 ft per min
Reduction ratio	—	36:1
Percent reduction	—	97.3

Only the alloy containing 20 pct Th was preheated for 16 hr, all other alloys were given a 2 hr preheat.

Table VII. Tensile Properties and Compressive Yield Strength of Extruded Mg-Th-Zr Alloys in the As-Extruded and in the Solution Heat-Treated Conditions at Room Temperature

Th, Pct	Acid Soluble Zr, Pct	As-Extruded				T4 Condition			
		Pct E*	TYS	CYS	TS	Pct E	TYS	CYS	TS
	0.69	11.7	27.6	11.6	38.2	5.6	19.3	5.8	32.7
0.93	0.82	36.0	13.8	13.2	26.4	23.6	7.3	6.2	22.0
1.84	0.82	38.0	14.4	13.6	27.0	27.6	7.8	6.8	23.1
3.34	0.94	34.0	16.0	15.8	27.5	26.0	8.5	8.2	24.4
6.05	0.94	30.0	18.5	18.6	29.3	30.6	10.5	11.0	26.2
10.30	0.98	22.4	21.7	23.0	31.8	26.0	13.4	14.0	28.2
19.70	1.01	11.3	27.2	29.6	36.1	12.0	18.4	19.0	29.0

* Pct E = Percent elongation in 1.5 in.
CYS = Compressive yield strength in 1000 psi.

Table VIII. Tensile Properties and Compressive Yield Strength of Extruded Mg-Th-Ce-Zr Alloys

Th, Pct	Ce, Pct	Acid Soluble Zr, Pct	As-Extruded				T5 Condition											
			Room Temperature				Room Temperature				400°F			500°F			600°F			700°F		
			Pct E*	TYS	CYS	TS	Pct E*	TYS	CYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS
1.34		0.94	34.0	16.0	15.8	27.5	28.6	18.5	18.6	33.3	34.4	15.2	19.8	34.0	13.6	17.6	66.7	9.1	12.4	120.0	4.4	7.4
1.37	1.25	0.97	25.3	17.3	16.8	28.6	21.3	20.6	19.4	34.8	27.0	16.2	21.7	32.0	13.8	21.3	67.6	9.5	14.4	154.5	3.8	6.5
2.00	2.20	0.91	20.6	19.7	19.0	29.4	15.3	22.6	21.6	34.5	23.6	16.3	24.1	28.0	15.3	23.1	55.2	9.6	14.0	206.0	4.5	7.4
3.15	2.93	0.87	16.3	20.6	20.2	30.0	11.6	23.8	21.7	34.2	20.0	17.8	24.6	27.0	15.0	23.0	64.9	9.7	14.8	206.5	3.8	6.2
6.05		0.94	30.0	18.5	18.6	29.3	24.6	21.5	21.4	34.5	31.0	17.0	21.7	31.6	15.2	19.0	71.0	9.7	12.9	134.0	4.0	6.8

Th, Pct	Ce, Pct	Acid Soluble Zr, Pct	T4 Condition				T6 Condition											
			Room Temperature				Room Temperature				400°F			500°F			600°F			700°F		
			Pct E*	TYS	CYS	TS	Pct E*	TYS	CYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS	Pct E*	TYS	TS
1.34		0.94	26.0	8.5	8.2	24.4	18.7	11.7	11.1	31.5	31.6	10.2	20.7	34.3	9.9	19.8	19.0	9.2	16.2	52.7	4.4	8.0
1.37	1.25	0.97	26.6	11.8	11.2	26.8	20.0	14.9	14.6	34.1	27.0	12.8	24.9	29.6	12.2	23.6	39.6	10.0	17.3	102.0	4.2	7.6
2.00	2.20	0.91	23.0	13.1	13.2	27.3	17.0	16.4	16.4	34.0	23.3	13.4	26.4	23.0	13.5	25.1	44.0	10.6	18.2	104.9	4.4	7.8
3.15	2.93	0.87	18.6	14.2	13.9	27.6	12.3	17.8	16.9	34.6	18.0	13.9	25.5	20.6	13.2	25.0	42.3	10.8	18.6	80.3	5.1	8.6
6.05		0.94	30.6	10.5	11.0	26.2	22.7	13.9	13.2	34.7	29.0	11.8	23.0	29.3	11.4	21.6	16.0	10.7	16.8	17.7	7.0	11.5

* Pct E = Percent elongation in 1.5 in.

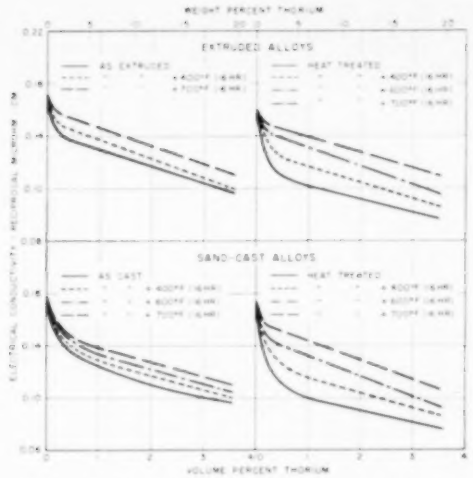


Fig. 10—Electrical conductivity of Mg-Th-Zr alloys at 95 F.

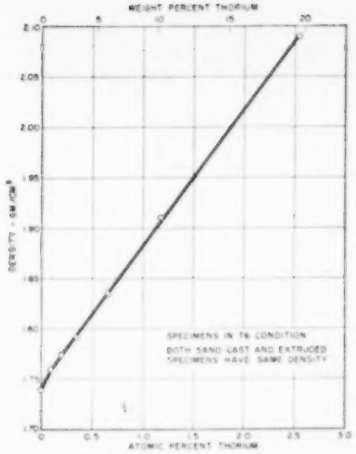

WEIGHT PERCENT THORIUM
SPECIMENS IN T6 CONDITION
BOTH SAND-CAST AND EXTRUDED SPECIMENS HAVE SAME DENSITY
ATOMIC PERCENT THORIUM

Fig. 11—Density of Mg-Th-Zr alloys at 77 F.

Tension, compression, and creep tests were performed in the same manner as described previously.[7]

The tensile properties of extruded Mg-Th-Zr alloys in the T5 and the T6 conditions are shown as a function of thorium content in Figs. 5 and 6, respectively. Extruded Mg-Th-Zr alloys exhibit some age hardening when aged either directly from the as-extruded condition or after solution heat treatment (cf. Table VII with Figs. 5 and 6). However, the degree of aging is not quite as great in the extruded alloys as that found in the sand-cast alloys of equivalent composition. The most striking feature to be observed in these curves is the sharp decrease in tensile strength and tensile yield strength of Mg-Zr alloy upon the addition of 1 pct Th. Further additions of thorium up to 20 pct result in a continuous increase of these properties. In contrast to this behavior, the compressive yield strength increases regularly with thorium content from 0 to 20 pct (Fig. 7). It should also be noted that addition of thorium either to magnesium or to Mg-Zr alloys results in equivalent tensile and compressive yield strengths in the extruded state. The present observations lead to the conclusion that thorium produces an orientation that is as favorable to plastic deformation under a tensile load as under a compressive load, inasmuch as the thorium decreases the tensile yield strength to a value equivalent to that of the compressive yield strength rather than the converse.

The ductility of Mg-Th-Zr alloys as measured by the elongation in a tensile test goes through a decided maximum value with increasing thorium content. The maximum occurs at a somewhat lower thorium content in the T5 condition than in the T6 condition. However, in both conditions, Mg-Th-Zr alloys exhibit an unusually high level of elongation over a wide range of thorium content.

The strength properties of these alloys at elevated temperatures increase with increasing thorium content from 0 to 20 pct Th. The pronounced decrease in strength of the Mg-Zr alloy with increasing temperature and the relative insensitivity of Mg-Th-Zr alloys to temperature eliminate the initial decrease in strength observed at room temperature. The presence of zirconium in extruded Mg-Th has no significant effect either on the room temperature or on the elevated temperature static properties of Mg-Th alloys.[7]

The curves in Figs. 8 and 9 depicting the change in creep limits of extruded Mg-Th-Zr alloys with thorium content show that the increase in creep resistance with thorium additions is proportionately

Table IX. Creep Limits of Extruded Mg-Th-Ce-Zr Alloys

			100-Hr Creep Limits (1000 psi)								
			400°F			500°F			600°F		
				Extension			Extension			Extension	
Th, Pct	Ce, Pct	Acid Soluble Zr, Pct	0.1 Pct Creep	0.2 Pct Total	0.5 Pct Total	0.1 Pct Creep	0.2 Pct Total	0.5 Pct Total	0.1 Pct Creep	0.2 Pct Total	0.5 Pct Total
T5 Condition											
3.34		0.94	10.8	10.3	13.4	4.1	4.2	5.1	1.4	1.5	1.7
3.37	1.25	0.97	13.2	11.0	14.1	4.4	4.6	6.4	1.6	1.6	1.8
2.90	2.20	0.91	14.0	11.5	14.8	3.9	4.1	6.0	1.0	1.2	1.7
3.15	2.93	0.87	13.6	11.7	14.2	3.5	3.7	4.4	1.2	1.3	1.6
6.05		0.94	13.2	10.7	14.1	4.1	4.2	6.0	1.8	1.9	2.2
T6 Condition											
3.34		0.94	15.0	8.7	12.0	5.8	5.3	9.0	1.3	1.8	2.6
3.37	1.25	0.97	14.2	10.5	15.0	6.6	6.4	7.8	1.3	1.5	1.7
2.90	2.20	0.91	14.0	11.5	14.0	6.2	6.2	7.7	1.5	1.6	2.0
3.15	2.93	0.87	14.0	10.0	13.5	5.1	5.3	6.9	1.6	1.8	2.3
6.05		0.94	13.0	10.0	13.0	8.0	7.6	9.4	1.9	2.0	2.6

greater at 400° and 500°F than at 600°F. The T5 condition shows some advantage over the T6 condition at 400°F particularly in the lower thorium contents. At 500°F, however, the T6 condition is decidedly superior to the T5 condition but not to the extent that was observed in binary Mg-Th alloys.[1] The results of the tests at 600°F show that the T5 and T6 conditions are equivalent in creep resistance.

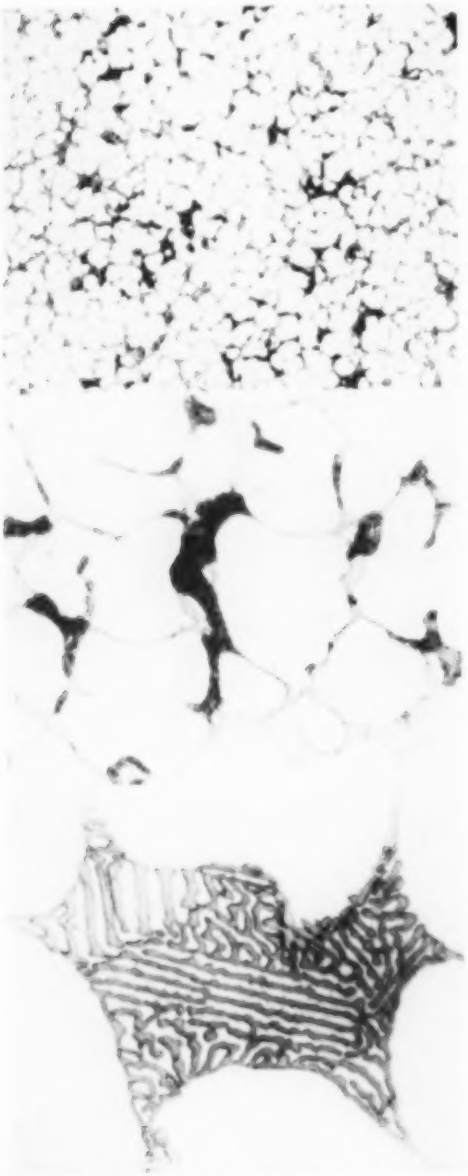


Fig. 12—Structure of sand cast Mg-1.9 pct Th-1.01 pct Zr in the as-cast condition. Glycol etchant. a (top)—General structure. X100. b (center)—Eutectic and compound particles. X500. c (bottom)—Lamellar structure of eutectic. X2000.

This is contrary to previous findings on Mg-Th alloys[1] and on Mg-mischmetal-Mn alloys,[4] in which the creep resistance at 600°F in the T5 condition is markedly lower than that in the T6 condition.

As for the effect of zirconium on the creep resistance of Mg-Th alloys, the most significant changes to be observed are the marked increases in creep limits at 500° and 600°F in the T5 condition, although some improvement is realized also in the T6 condition at 400° and 500°F. There obviously remains a great deal of work of a fundamental nature to be done in order to arrive at a better understanding of the effect of the various alloying elements on the elevated-temperature creep resistance of magnesium alloys.

Some improvement in strength of extruded Mg-Th-Zr is obtained from the addition of pure cerium (Table VIII). However, the increases are relatively small and are realized, for the most part, at room temperature. The effect of cerium on the creep resistance of these alloys is even less significant (Table IX). The most significant change occurs at 500°F where cerium exerts a definitely detrimental effect on the creep limits, particularly in the T6 condition.

Sauerwald[5] does not present any data on either Mg-Th-Zr or Mg-Th-Ce-Zr alloys that can be compared directly with the present results. Somewhat higher creep resistance is indicated by Sauerwald's data on a Mg-Th-Ce-Zr alloy than that obtained on the corresponding composition in this investigation. However, although it is not specifically stated by Sauerwald, it is believed that his alloy contained mischmetal instead of pure cerium as was used in our alloys. Furthermore, the alloy was tested by Sauerwald in the as-extruded condition, whereas the present results were obtained in the aged or in the heat-treated plus aged condition.

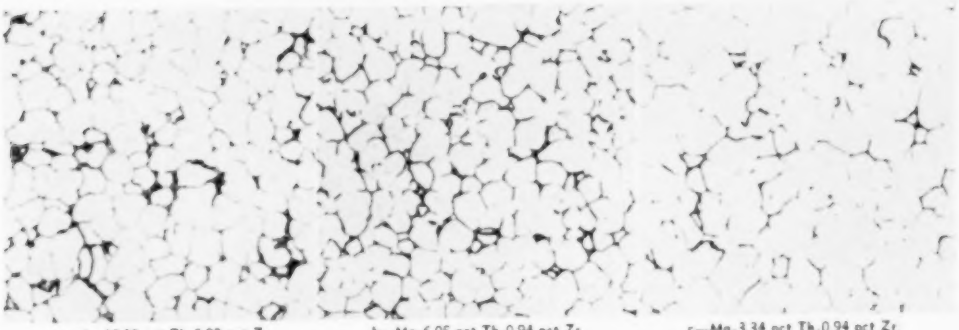
Physical Properties

Electrical conductivity and density measurements were conducted in the same manner as described earlier.[1] Addition of zirconium lowers the conductivity of Mg-Th alloys (Fig. 10) significantly (see ref. 1 for comparison). In the composition range where most of the thorium is present as an intermetallic compound, the conductivity decreases linearly with increasing volume pct thorium (Fig. 10). These data indicate somewhat different solubility relationships than those found in the binary Mg-Th alloys. The maximum solid solubility as shown by the conductivity curves for the heat-treated condition indicates a somewhat lower maximum solubility of thorium in the Mg-Zr than in magnesium whereas the curves obtained after aging at 700°F lead to a greater solubility at that temperature than obtained in the binary alloys. Considerably more work is needed to establish solubility and phase relationships in these alloy systems.

The change in density of Mg-Th-Zr alloys as a function of thorium content is given in Fig. 11. No significant difference exists between the density of sand-cast and extruded alloys.

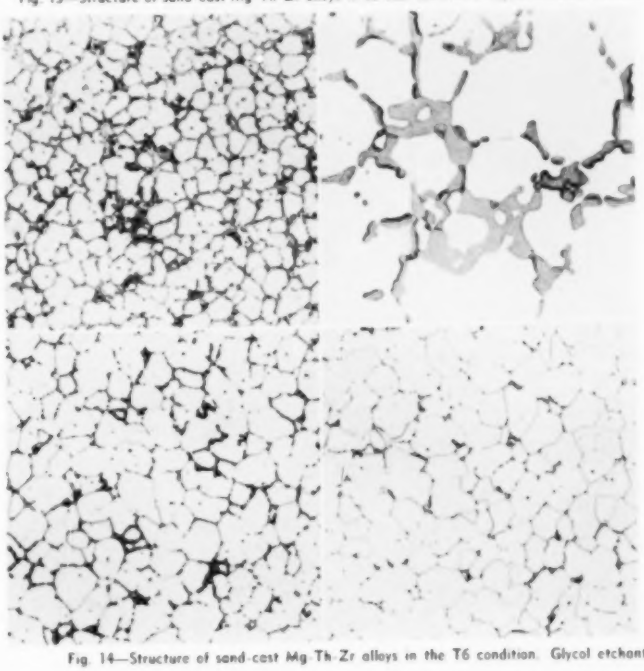
Metallography

A few micrographs are presented to illustrate the nature of the microstructure of Mg-Th-Zr and Mg-Th-Ce-Zr alloys. The fine-grain nature of the structure of sand-cast Mg-Th-Zr is clearly shown by the micrographs at 100 magnification (Figs. 12a and 13a, b, and c). The grain boundaries are well delineated by the eutectic or the free intermetallic compound resulting from divorcement of the eutectic. The pres-



a—Mg-10.30 pct Th-0.98 pct Zr. b—Mg-6.05 pct Th-0.94 pct Zr. c—Mg-3.34 pct Th-0.94 pct Zr.

Fig. 13—Structure of sand-cast Mg-Th-Zr alloys in as-cast condition. Glycol etchant. X100



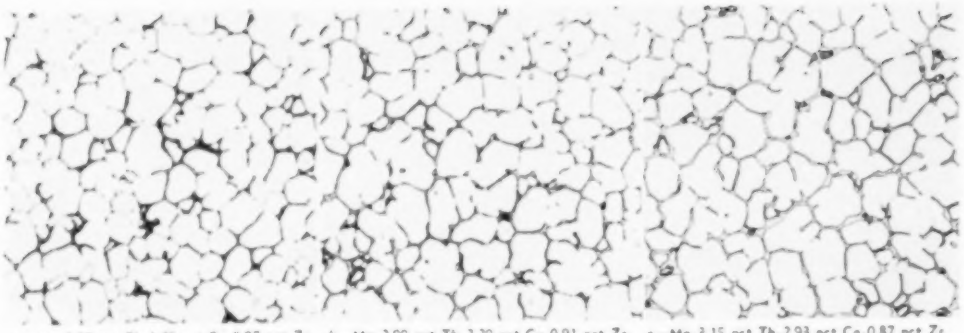
a—Mg-19.70 pct Th-1.01 pct Zr. X100. b—Mg-19.70 pct Th-1.01 pct Zr. X500. c—Mg-10.30 pct Th-0.98 pct Zr. X100. d—Mg-6.05 pct Th-0.94 pct Zr. X100.

Fig. 14—Structure of sand-cast Mg-Th-Zr alloys in the T6 condition. Glycol etchant.

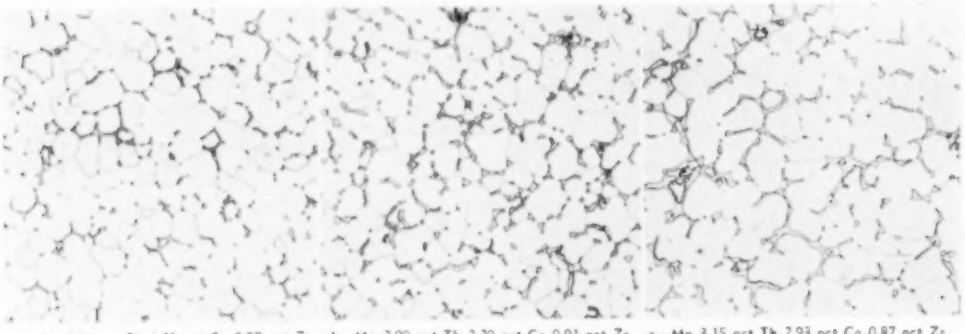
ence of both eutectic and compound is shown for the Mg-20 pct Th-1 pct Zr alloy in Fig. 12b. The fine eutectic has the same lamellar structure (Fig. 12c) that was noted in binary Mg-Th alloys.[4] The decreasing amount of compound and eutectic with decreasing thorium content is shown by the series of micrographs in Fig. 13, where the structures of alloys containing 10, 6 and 3 pct Th are illustrated. Small amounts of eutectic were also observed in alloys containing 2 and 1 pct Th.

The structures in Fig. 14 show that heat treatment effects considerable solution of compound and the remaining undissolved compound is well coalesced. Two distinct compounds are present in the Mg-20 pct Th-1 pct Zr alloy (Fig. 14b), as has been observed in binary Mg-Th alloys containing 20 pct Th or more.[5] The amount of undissolved compound decreases rapidly with thorium content (Figs. 14c and d). Structures of alloys containing less than 6 pct Th are not shown, but they were found to contain only traces of undissolved compound.

Addition of cerium markedly increases the amount of intermetallic compound in Mg-3 pct Th-1 pct Zr alloy (Fig. 15) as would be expected from the relatively low solid solubility of cerium in magnesium. Another distinguishing feature of the structure of the cerium-containing alloys is the high degree of divorcement of the eutectic in alloys containing 2 and 3 pct Ce. This structure is typical of magnesium alloys containing mixtures of rare-earth metals;[6] combining thorium with cerium apparently has the same effect. The large increase in compound resulting from cerium additions is more clearly illustrated by the structures of the T6 condition (Fig. 16) where the pronounced increase in the amount of undissolved compound is apparent; it was pointed out above that the Mg-3 pct Th-1 pct Zr contains only traces of undissolved compound after heat treatment. The



a—Mg 3.37 pct Th 1.25 pct Ce 0.97 pct Zr. b—Mg 2.90 pct Th 2.20 pct Ce 0.91 pct Zr. c—Mg 3.15 pct Th 2.93 pct Ce 0.87 pct Zr.
Fig. 15—Structure of sand cast Mg-Th-Ce-Zr alloys in the as cast condition. Glycol etchant. X100.



a—Mg 3.37 pct Th 1.25 pct Ce 0.97 pct Zr. b—Mg 2.90 pct Th 2.20 pct Ce 0.91 pct Zr. c—Mg 3.15 pct Th 2.93 pct Ce 0.87 pct Zr.
Fig. 16—Structure of sand cast Mg-Th-Ce-Zr alloys in the T6 condition. Glycol etchant. X100.

cerium-containing alloys also develop considerably more fine precipitate in the center of the grains upon heat treatment than is observed in Mg-Th-Zr alloys, in fact in the latter it is hardly detectable, particularly in alloys containing more than 3 pct Th. This precipitate has been demonstrated by Nelson and Strieter[7] to form in Mg-mischmetal-Zr alloys and evidence has been presented by Mellor[8] showing that it consists principally of zirconium-hydride and some metallic zirconium.

Summary

The results of this investigation show that the addition of zirconium to sand-cast Mg-Th alloys contributes an unusual combination of desirable properties. As in many magnesium alloy systems, zirconium markedly reduces the grain size of Mg-Th alloys; in fact, the characteristic, coarse columnar structure of the binary alloys is changed to one consisting of fine equiaxed grains of 0.001 to 0.002 in. diam.

The decrease in grain size is accompanied by a significant increase in mechanical properties over the entire range of thorium content investigated. The beneficial effect of zirconium on the strength properties is maintained at elevated temperatures up to 700 F. Furthermore, zirconium increases the creep resistance of Mg-Th alloys at 400 and 500 F, whereas it produces little, if any, change on the creep resistance at 600 F. The result of these effects is that sand-cast Mg-Th-Zr alloys are the only magnesium alloys found to date having the desirable combination of fine grain size and good creep resistance at temperatures up to 600 F. The optimum thorium content appears to be 3 pct; more work is required to establish the preferred amount of zirconium.

The addition of zirconium does not produce any great improvement in either the strength or creep characteristics of extruded Mg-Th alloys. The most striking feature of extruded Mg-Th-Zr alloys is the equivalence in the creep resistance at 600 F in the T5 and T6 conditions.

Cerium is not a desirable addition to Mg-Th-Zr alloys.

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Optical Temperature Scale and Emissivities Of Liquid Iron-Copper-Nickel Alloys

by David B. Smith and John Chipman

THIS investigation was undertaken as a prerequisite to the study of sulphur activities in the liquid system Fe-Cu-Ni, a continuation of the work of Sherman, Elvander, and Chipman,[1] using the same equipment and the same experimental technique.

Temperature measurements are made with a disappearing filament optical pyrometer, sighting through a prism onto the surface of the melt which is heated by induction. To obtain true temperatures by this means, a calibration is necessary, since, in the absence of true black-body conditions, the observed temperature is always lower than the true temperature. This calibration is actually an emissivity correction, since the emissivity is a function of both temperature and melt composition.

The original furnace was used with a deeper metal bath to provide sufficient depth of immersion for the thermocouple which was inserted through the sampling hole. The atmosphere was a 50-50 mixture or argon and hydrogen passing through the furnace at a rate of 1000 ml per min, and preheated by means of a molybdenum resistance coil. It was assumed that the rapid gas flow effectively eliminated any error which might have resulted from the presence of metallic vapor. The charge in each case consisted of about 300 g of metal, contained in a high purity, dense alumina crucible.

At each melt composition, readings of the optical pyrometer and of a thermocouple immersed in the molten metal were taken simultaneously. Calibration curves of optical vs. true temperature were then made for each melt composition investigated.

Thermocouples were made of 0.010 in. platinum and platinum-10 pct rhodium. They were checked against the melting point of chemically pure copper. Silica protection tubes with an outer diameter of approximately $\frac{1}{8}$ in. were used. The depth of immersion of the tip in the molten metal was approximately $1\frac{1}{4}$ in. The life of these small thermocouples was relatively short, varying from approximately 1 min at 1550°C to 10 min or longer at 1300°C. Melts containing iron corroded the protection tubes more than those without iron present.

Measurements taken while heating checked those taken while cooling, indicating the absence of a temperature gradient between the molten metal and the thermocouple.

Results

The temperature range of the measurements on copper-free metals extended from the melting point to 1600°C. For copper the upper limit was 1380°C, and the data were extrapolated linearly for comparison with observations on the other metals and alloys at 1535°C.

Examples of typical calibration curves for various melt compositions are shown in Fig. 1. These curves, besides providing a calibration system for the given experimental conditions, also give a basis for the calculation of true emissivities at the effective wavelength of the pyrometer, 0.65 micron.

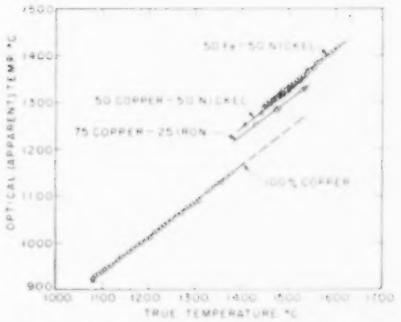

Fig. 1—Optical vs. true temperatures for typical compositions.

The starting point of the calculations is the value of the emissivity of iron at its melting point. Taking the melting point of electrolytic iron under hydrogen as 1535°C, and using the corresponding emissivity value of 0.43, as determined by Dastur and Gokcen,[2] the emissivities can be determined as functions of composition (and temperature, if desired).

The relationship used is the Wien equation:

$$\ln(E_\alpha) = \frac{C_2}{\lambda}\left(\frac{1}{T_t} - \frac{1}{T_a}\right)$$

where E is the emissivity; α, the transmissivity of the optical system; C_2, the fundamental constant, 14,380 micron-degrees; λ, the wave length of light used (0.65 micron); T_t, the true temperature, °K; and T_a, the apparent temperature, °K.

At the melting point of iron the observed temperature was 1358°; this gives 0.62 for the transmissivity, which compares with 0.65 in the similar system used by Dastur and Gokcen. The emissivities of all the compositions investigated were determined at 1535°C, after obtaining from each calibration curve the corresponding temperature. These results

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Fig. 2—Emissivity of Fe Cu alloys at 1535°C.

Fig. 3—Emissivity of Ni Cu alloys at 1535°C.

Fig. 4—Emissivity of Fe Ni alloys at 1535°C.

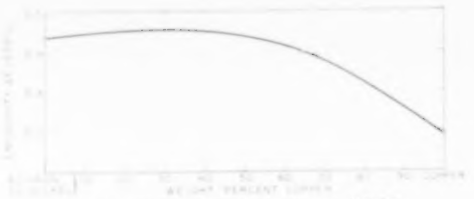

Fig. 5—Emissivity of Fe Ni Cu alloys at 1535°C.

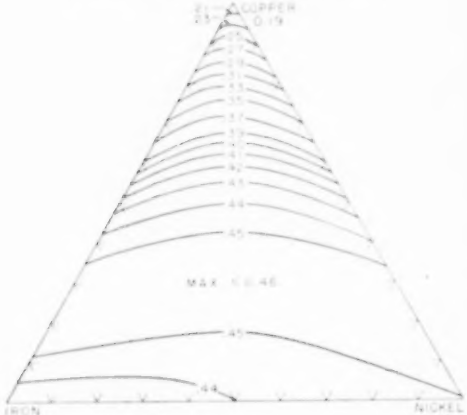

Fig. 6—Lines of constant emissivity at 1535°C.

are listed in Table I and shown graphically in Figs. 2 to 5.

Discussion of Results

Roeser and Wensel[1] listed the following emissivity values for molten metals: Cu, 0.15; Fe, 0.37; and Ni, 0.37. Our results, like those of Dastur and Gokcen are somewhat higher. We have used their datum for iron as the basis for computation so as to have a consistent set of values for the ternary system. If this datum is in error, our results will require proportional correction. A part of the difference may be due to the fact that in each case a gas preheater was used (in order to duplicate conditions to be used in later work). However, the effect of the preheater on optical readings was not great enough to account for more than a small part of this difference.

The data are shown in the form of a ternary plot in Fig. 6. In view of the simplicity of the Fe-Ni plot, and the similarity of the other three curves, the lines of the ternary are readily constructed.

In each of the binary systems containing copper, a maximum emissivity is found at about 20 to 30 pct Cu. The maximum deviation from the simple law of mixtures is found at about 50 to 60 pct Cu. It seems possible that this is due to the electronic effect discussed by Mott[6] on the basis of the work of Lowery, Bor, and Wilkinson.[7] This work showed a maximum absorbtion coefficient in solid Cu-Ni solutions at roughly the same percentage of copper as the maximum in emissivity found in this investigation.

Table I. Apparent Temperatures and Emissivities of Liquid Metals at 1535°C (Wavelength, 0.65 micron)

Composition Wt Pct			Apparent Temperature, °C	Emissivity E
Cu	Fe	Ni		
100			1267	0.19
75	25		1329	0.28
60	40		1343	0.38
54	46		1353	0.42
52	48		1352	0.41
45	55		1353	0.42
40	60		1354	0.43
25	75		1368	0.47
22	78		1358	0.43
	100		1363	0.45
		100	1360	0.44
	50	50	1352	0.41
50		50	1332	0.34
75		25	1339	0.36
25		75	1365	0.46
33.33	33.33	33.33	1365	0.46
60	10	30	1347	0.39
64.8	17.78	17.78	1349	0.40

Acknowledgments

The authors wish to thank L. Ticknor, F. Irmann, and N. Gokcen, for their assistance in temperature readings. This investigation forms a part of a study of the thermodynamics of metallic solutions sponsored by the Atomic Energy Commission under Contract No. AT-30-GEN-1002.

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Transformation in Cobalt-Nickel Alloys

by J. B. Hess and C. S. Barrett

TO reach equilibrium between different phases in cobalt-rich alloys requires prohibitively long annealing times when temperatures are below about 700°C. The fact that a transformation from face-centered cubic to close-packed hexagonal readily takes place at temperatures below this in the cobalt-rich solid solutions is not an indication that thermally activated processes occur at an appreciable rate, for the transformation is well established as martensitic in nature. Wide divergence between heating and cooling experiments and high sensitivity to prior heat treatment make it difficult to judge temperatures of equilibrium between the phases.[1-3] One object of the present work was to see if the information on the relative stability of phases could be gained by substituting plastic deformation for thermal agitation. Procedures were worked out that led to the determination of a diffusionless type of phase diagram, which represents the temperature of equal stability for phases of the same composition, and the technique was applied to the Co-Ni system. Another object of the work was to see whether or not deformation would generate frequent stacking faults when these were thin lamellae of a phase having higher free energy than the parent phase.

The alloys were prepared in 80 to 100 g melts from cobalt (with metallic impurities estimated spectrochemically as follows: Ni, 0.05 pct; Fe, 0.001 pct; Mg, Si, Cu, Cr, Al, < 0.001 pct) and Mond Carbonyl nickel (with Fe, 0.05 pct; Si, 0.003 pct; C, 0.61 pct; Cu, 0.001 pct; Co, Cr not detected, < 0.01 pct). The metals were melted in pure Al_2O_3 crucibles. An atmosphere of argon, that had been purified by passing over hot magnesium chips, was used for the alloys that, by analysis of the portions of the ingots actually used, were found to contain 15.3, 25.7, and 35.0 pct Ni, and vacuum melting (after degassing) was used for those containing 29.4 and 31.5 pct Ni. After induction melting the alloys were allowed to solidify in the crucible, and slices $^3/_8$ in. thick x $^1/_2$ in. in diam were annealed 12 hr at 1350°C for homogenization. These same specimens were used throughout the series of experiments, with annealing treatments of 4 hr at 900°C in purified hydrogen followed by furnace cooling, alternating with the deformation and X-ray tests discussed below.

Results

Spontaneous transformation was observed on cooling to room temperature in all alloys containing 29.4 pct Ni or less and by cooling the 31.5 pct alloy to −195°C but was not observed in the 35 pct alloys after cooling to −195°C. These results are in satisfactory agreement with the cooling experiments of Masimoto.[4]

From these data it is clear that the temperature of beginning transformation M_s drops to 20°C with the addition of about 30 pct Ni. The test for spontaneous transformation was metallographic. Specimens were thermally polished by annealing 10 hr in hydrogen at 1350°C, then furnace cooled; if transformation had occurred there were relief effects visible on the thermally polished surfaces. These markings were narrow straight lines, usually resolvable at high magnification as clusters of fine lines that resembled slip lines. It was concluded that they resulted from displacements on {111} planes, for the number of directions in individual grains often reached but never exceeded four, and lines could always be found parallel to the thermally etched {111} boundaries of annealing twins. The markings were thus consistent with the idea that the transformation occurs by (111) plane displacements (Shockley partial dislocations moving on (111) planes). This was further confirmed by X-ray tests for stacking disorders. Using an oscillating crystal technique previously employed to detect strain-induced faulting in Cu-Si alloys,[5] streaks indicative of the stacking faults were looked for and found on X-ray films of the spontaneously transformed 25.7 pct Ni alloys, as expected by analogy with Edwards and Lipson's results on pure cobalt.[6] The streaks were much intensified after rolling at room temperature.

Transformation induced by plastic strain was investigated as a function of alloy composition and temperature of deformation. A series of tests was made to determine suitable straining and X-raying techniques. Filing was found inferior to abrasion in converting cubic samples to hexagonal, and abrasion was less effective than peening in producing smooth unspotty Debye rings in the X-ray patterns. Because the diffraction lines were broad, Geiger-counter spectrometer records of filings were less sensitive in revealing small amounts of transformed material than X-ray patterns recorded on films in a small diameter camera. After these exploratory tests the following methods were adopted. Specimens that had been annealed at least 4 hr at 900°C and furnace cooled were mounted in a block of aluminum, brought to temperature, and peened thoroughly with a mullite pestle preheated to the same temperature. The specimens were then quenched to room temperature. In peening, a circular area of $^3/_8$ in. diam was given 500 blows. A few control tests showed that an additional 1000 blows did not detectably change the proportions of the phases present.

The amount of transformation was judged by X-ray reflection patterns from the peened surface, using the innermost four lines of the cubic and the hexagonal patterns with filtered $CoK\alpha$ radiation,

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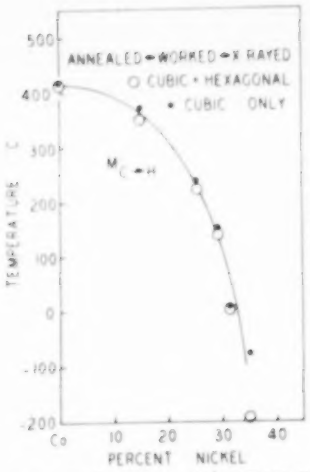

Fig. 1—Strain transformation of cubic phase to hexagonal in Co-Ni alloys. Curve shows highest temperature at which deformation causes detectable strain transformation in previously annealed samples.

in an oscillating crystal camera operating at room temperature. The Debye rings obtained after this treatment were invariably smooth and free from spottiness, indicating thorough cold working of the grains.

The results are presented in Fig. 1, where a curve is drawn through the data. Specimens in the annealed state that were worked at temperatures below this curve invariably contained at least a trace of hexagonal material that was produced by the straining; the curve thus gives temperatures that are often referred to as M_s temperatures, and can be called the M_s curve. The amount of the hexagonal increased as the working temperature was lowered 10° to 25° C below the curve and increased further at lower working temperatures. However, complete strain transformation to hexagonal was never found, even when pure cobalt was peened at a temperature more than 400° C below M_s.

The method of X-raying at room temperature after deforming at elevated temperatures could be used to establish M_s temperatures because of the fact that working tended to stabilize the samples against spontaneous transformation on subsequent cooling. Cobalt and the 15.3 pct Ni alloy could not be cooled to room temperature without some hexagonal forming even when previously peened above M_s, so it was necessary to compare this irreducible amount of hexagonal with the increasing amounts that were obtained by working at lower temperatures; for alloys of higher nickel content, however, peening above M_s fully suppressed the transformation on subsequent cooling to room temperature.

In addition to finding this stabilizing effect of cold work on spontaneous transformation, an analogous inhibiting effect was found for the strain-induced transformation from cubic to hexagonal. For example, some specimens that had been worked above M_s showed no hexagonal lines after additional peening at temperatures as much as 60° below M_s. It was because of this effect that the M_s curve was established with alloys that had been annealed prior to each hot working experiment.

The reverse transformation was studied by first peening at a low temperature to form a large amount of hexagonal material, X-raying, then peening at higher temperature, and again X-raying. The second peening (with no intermediate annealing) caused reversion of some of the hexagonal to cubic whenever the temperature of the second deformation was sufficiently high.

Fig. 2 shows the minimum deformation temperature for this reversion to begin. The curve is bracketed in a temperature interval of about ±12° C, and by comparison with Fig. 1 it will be seen that within this range of accuracy the curve of Fig. 2 coincides with the curve of Fig. 1. Thus plastic flow in annealed samples below M_s produces the hexagonal phase, in limited amount, from the cubic, and plastic flow above M_s begins the reversion of this hexagonal to cubic. Raising the deformation temperature increases the extent of reversion from hexagonal to cubic until complete reversion is accomplished. The temperature at which complete reversion is possible in each alloy was not accurately determined—it may, in fact, depend strongly on the amount of deformation—but appeared to lie in the range 10° to 60° above M_s with the technique employed here.

The M_s temperature for pure cobalt was determined in the present tests as lying between 412° and 427° C and strain reversion to cubic was detected at 421° C in a sample previously cold worked at room temperature, these temperatures having an uncertainty of ± 2°. From these data it is concluded that the transformation point for cobalt when being plastically deformed is between 412° and 421° C and may be taken as 417° ± 7° C. Previously published results indicate that the equilibrium temperature for the two phases is above 388° C, the M_s temperature in large grained cobalt, and below the temperature for the transformation on heating, which is often reported to be 440° to 450° C; the equilibrium temperature has been estimated by some investigators to lie in the range 420° to 430° C.[5]

Discussion

Reversion to the cubic structure can occur merely by the reverse movement of the same partial dislocations that brought about conversion to the hexagonal, or equivalently, by passage of one additional partial dislocation (or an odd number of them) on alternate basal planes. Or if some rotary dislocation movement is involved, as has been suggested for twinning,[7] the reversion and conversion processes also would involve reverse directions of motion. In both phases the planes traversed by the transformation dislocations are slip planes. From consideration of these facts and also of the marked similarity of the two phases and the low energy required to create a coherent interface between them, it seems probable that the activation energies that must be overcome in the conversion and reversion processes are equal at the temperature for which the free energies of the cubic and hexagonal phases are equal. At higher temperatures the barrier that has to be surmounted by a cubic-to-hexagonal dislocation movement should be greater than the barrier for reversion, hence any stresses capable of initiating the cubic-to-hexagonal movement should be more than enough to surmount the barrier for return to cubic. At lower temperatures the situation is reversed. In the absence of disturbing factors, this interpretation means that the temperature at which the two phases have equal free energies lies between the temperatures of beginning strain-induced conversion and beginning strain-induced reversion, temperatures that are indicated by the curves of Figs. 1 and 2, respectively, for Co-Ni alloys, and that to an accuracy of about ± 12°, the M_s

curve can be taken as the curve for equal free energy of the two phases as they exist in the sample at the beginning of conversion and reversion. The two phases have the same composition, of course, since the transformations are diffusionless. The assumption that disturbing factors are absent requires discussion. Actually, the alloys used for tests of reversion to cubic had previously been heavily cold worked and, on the other hand, those used for conversion to hexagonal had been annealed. It might be imagined, therefore, that the reversion process was more subject to hindrance from dislocations and lattice distortions than the conversion process. Nevertheless, the fact that reversion began at very nearly the same temperature as conversion did, and not at a definitely higher temperature, suggests that this factor was unimportant in the present tests.

A striking fact that requires explanation is that plastic flow at any temperature never converted all cubic metal to hexagonal, whereas flow at temperatures somewhat above M_s but still below recrystallization temperatures reverted all hexagonal metal to cubic (insofar as can be judged from the films of the higher nickel alloys). This unsymmetrical behavior may be accounted for on the basis of relative rates of strain hardening of the two phases. If it is assumed that the cubic phase strain hardens more rapidly than the hexagonal, it would follow that flow below M_s would concentrate to an increasing degree in the hexagonal as straining proceeded, and would consequently become progressively less effective in converting the remaining cubic metal. At temperatures above M_s, a similar concentration of flow in the lesser hardening hexagonal phase carries the transformation of this phase to completion. That the cubic phase strain hardens more rapidly than the hexagonal would be expected from the well-known tensile stress-strain curves for single crystals of face-centered cubic metals slipping on intersecting planes as compared with the curves for close-packed hexagonal metals slipping only on the basal plane.

The effect of prior plastic flow above M_s in inhibiting both spontaneous and strain-induced transformations to the hexagonal phase is understandable in terms of interference to the movement of partial dislocations. In annealed metal there is microscopic evidence that the hexagonal plates have diameters that are comparable with the diameters of the cubic grains. The partial dislocations must therefore travel large distances without encountering effective barriers. In the worked metal, however, they must encounter frequent barriers in the form of intersecting dislocations and opposing microstresses.

One object of the present work was to determine whether appreciable numbers of stacking faults can be introduced into face-centered cubic metal when it is deformed at a temperature where it is more stable than close-packed hexagonal metal of the same composition. Accordingly the attempt was made to detect faults in Co-Ni samples lightly deformed at various temperatures. The experimental technique was that used previously for fault detection in polycrystalline Cu-Si alloys,[7] a search for non-Laue streaks in oscillating crystal X-ray diffraction patterns of lightly deformed samples. In the alloy with 31.5 pct Ni, the fault streaks were detected after 0.5 pct reduction by rolling at room temperature and after deformation at liquid nitrogen temperature, but were not found after various deformation tests at 43°C in this alloy nor after room temperature deformation in the 35.0 pct alloy.

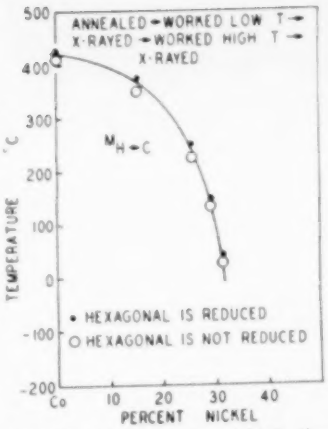

Fig. 2—Strain transformation of hexagonal phase to cubic in Co-Ni alloys. Curve shows lowest temperatures at which deformation causes detectable hexagonal to cubic transformation in alloys previously strained at low temperatures.

Although the accuracy of the test is not high, the results indicate that a temperature very near M_s is the highest temperature at which deformation produces large amounts of faulting, and therefore that strain cannot induce much faulted material when the cubic phase is more stable than the hexagonal. The stability referred to here is stability during mechanical flow, but the reasoning presented earlier suggests that it also applies to the free energy of the phases; in other words, it appears that for the most part half-dislocations cannot dissociate and separate widely, creating a faulted region between them which is a thin layer of hexagonal structure, if this structure is of appreciably higher free energy than the parent cubic structure.

Summary

A curve was determined showing the temperatures at which deformation begins to transform the cubic phase to the hexagonal phase in cobalt-rich Co-Ni alloys. A similar curve was determined for the strain-induced reverse transformation in the same alloys. The two curves agreed within experimental error and thus determine a curve of equal stability, which is also judged to be the curve giving equal free energy for the phases when they have the same composition. Stacking faults were detected after deformation at temperatures below this curve and not at temperatures definitely above it. The transformation temperature for strain-induced transformation in cobalt of 99.95 pct purity is 417° ± 7°C.

Acknowledgments

We are pleased to acknowledge the assistance of F. D'Heurle and W. Bergmann in the preparation and treatment of these alloys.

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Effect of Prior Strain at Low Temperatures On the Properties of Some Close-Packed Metals At Room Temperature

by W. C. Ellis and E. S. Greiner

WHEN metallic materials are deformed plastically, the process may be considered as one in which hardening and recovery occur simultaneously. The net hardening is that produced by deformation in the absence of recovery lessened by recovery during and immediately following deformation. Strain hardening takes place through movements of large groups of atoms acting to a degree as an entity. A complex microstress system is developed. Recovery is a diffusive-type process involving single atoms or, at most, only a few atoms in concerted action. This is accompanied by elastic strain relaxation. Recovery is strongly temperature-dependent. The driving energy for the process resides in thermal fluctuations of the atoms augmented by the energy of the stress field producing the deformation, and by that produced by the deformation. At very low temperatures recovery is essentially absent, or very small. At high temperatures in the hot working range, recovery is so rapid that deformation results in no appreciable net hardening.

The interplay of strain-hardening and recovery results in differing end structures depending on the temperature and the strain rate of the process. For example, there is a prominent difference in the spacing of the slip markings and in the detail of X-ray diffraction patterns. High temperatures and slow strain rates favor a wide spacing of slip markings and sharp reflections.[1] This suggests that change in rate of the recovery concurrent with deformation modifies to a degree the manner of the deformation. Different slip systems may operate. For very slow rates, the mechanism may, as has been suggested, differ from that of "classical slip." In the experimental work that follows the findings will be interpreted from the point of view developed in this section.

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Recovery and Electrical Conductivity

Electrical conductivity is a sensitive indicator of the state of strain in a material[1-3] and was used in this investigation. The materials studied in wire form were: OFHC copper, a high purity (99.99 pct) and 2S grade aluminum, and grade A nickel. Wire annealed and initially 0.050 in. in diam was drawn through diamond dies in a simple holder maintained at 25°, −75°, or −195° C. Precautions were taken that for each material all conditions of drawing were the same except the temperature. Electrical resistivity was measured for each condition at 20° C. This was done as soon as practical after drawing to minimize possible room temperature recovery.

The data for the four materials are plotted in Fig. 1 in terms of the change in resistivity for drawing at the several temperatures. The effect of temperature of drawing on the increase in resistivity is most pronounced for copper. Drawing at 25° C to 80 pct reduction in area increases the resistivity approximately 2 pct; drawing at −195° C to the same reduction increases the resistivity about 6 pct. The increase in resistivity[2-4] in this metal is due to extra scattering of conduction electrons originating in the lack of orderly arrangement of the atoms induced by the deformation process. It has been suggested

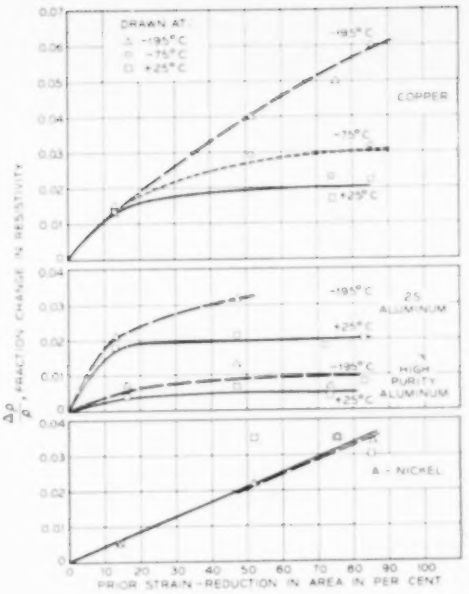

Fig. 1—Effect of temperature of drawing on resistivity at 20°C.

more specifically that the increased resistance is due to an elastic distortion produced by dislocations introduced by cold work.[4,5] Furthermore, it is reasonably certain that the resistivity measured is for the state after drawing—that there has been, at most, only an inappreciably small recovery at room temperature after drawing and prior to the measurement.

The effect of drawing for the two grades of aluminum is similar to that for copper, but of less intensity. Recovery at room temperature may have occurred to a degree in both purities of aluminum prior to measurement and quite probably did in the aluminum of the higher purity. This may be the chief reason for the difference in the observed resistivity increases for the two grades of aluminum.

For nickel erratic values were obtained, and no sustained difference in trend in resistivity change was found for drawing at 25° and −195°C. In nickel, a transition element, two factors contribute to electrical resistivity: scattering by the lattice, and to the *d*-band. The latter is the more important and for it, the effects of deformation, if any, have not been developed in adequate detail to be useful here.

Effect of Prior Strain at Low Temperatures on Strength Properties

The resistivity studies were supplemented by investigation of the effect on the strength properties of drawing wire and rolling sheet at low temperatures. The flow stress and other strength properties were measured in all cases at room temperature (approximately 25°C). Low temperature drawing increases the flow stress for a given strain as illustrated for copper in Fig. 2. Additional data for aluminum and nickel, supporting the same observation, are shown in Table I and in Fig. 3. The strength

Table I. Effect of Drawing at Low Temperature on Flow Stress and Strength at Room Temperature

Metal	Pct Reduction	Temperature of Draw, °C	Flow Stress, Psi at 0.005 Natural Strain	Ultimate Tensile Strength, Psi
Copper	51	25	52,000	55,000
		−195	59,500	67,000
High purity aluminum	16	25	13,800	14,900
		−195	17,000	16,000
2S aluminum	47	25	24,000	25,500
		−195	27,400	27,600
A-nickel	52	25	105,000	124,000
		−195	125,000	148,000

properties of nickel are enhanced by low temperature drawing whereas, as was discussed earlier, the temperature of drawing had little effect on the increase in resistivity. Others have observed an increase in flow stress in tensile tests on aluminum[6] and for cold-worked austenitic stainless steel.[7] The former was for tests made after prior strain in a tensile test at low temperature; for the austenitic stainless steels the strength increase is contributed to by a transformation induced by strain at the low temperature.

Rolling at low temperatures, as well as drawing, enhances the strength properties of metals at room temperatures. This is illustrated in Table II giving data for a number of common materials, some of which are alloys, processed at −195° and at 25°C. The large effects are in ultimate tensile strength, yield strength, ductility, and hardness—the plastic

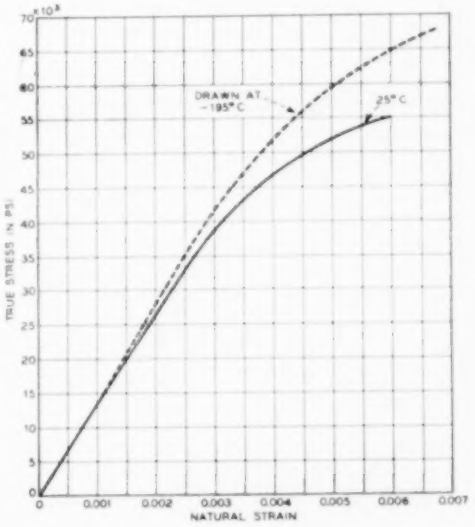

Fig. 2—Flow stress curves for copper at room temperature—prior strain, 51 pct reduction in area.

Table II. Effect of Rolling Temperature on Strength Properties of Some Metallic Materials

Material	Reduction in Thickness by Rolling, Pct	Rolling Temperature, °F	Ultimate Tensile Strength, Psi	P-Limit, Psi	Yield Strength, 0.01 Pct Set, Psi	Yield Strength, 0.2 Pct Set, Psi	Modulus of Elasticity, Psi x 10^{-6}	Elongation, Pct in 2 In.	Rockwell Hardness	
99 Al (R)*	37.5	25	17,000	8,200	9,800	16,200	7.9	6.0	53.3	"15T" scale
99 Al (R)	37.5	−195	18,700	9,200	11,200	18,300	8.9	2.5	57.7	
Copper (R)	37.5	25	47,400	29,500	33,800	46,200	14.5	4.0	53.6	"B" scale
Copper (R)	37.5	−195	54,600	31,700	39,100	53,800	14.3	2.1	60.9	
"A" brass (R)	49	25	84,000	32,500	46,000	73,600	13.1	5.1	88.3	"B" scale
"A" brass (R)	49	−195	96,100	35,700	48,800	82,000	12.7	1.8	92.4	
"A" phos. bronze (R)	23.4	25	81,800	46,100	55,300	76,600	14.6	6.9	88.9	"B" scale
"A" phos. bronze (R)	23.4	−190	92,500	48,700	56,200	87,200	16.5	3.5	93.0	
B Ni Ag (R)	37.5	25	100,900	46,300	66,200	97,400	19.0	2.6	96.1	"B" scale
B Ni Ag (R)	37.5	−190	109,400	43,700	62,700	105,600	18.3	1.9	97.7	

* Number of samples tested.

properties. There is no large effect on the proportional limit or on the modulus of elasticity, both of which are elastic properties.

Discussion of Results

The authors attribute the increase in resistivity and flow stress for a given strain after low temperature deformation to a smaller contribution from simultaneous recovery as the temperature of deformation is lowered. Recovery is reasonably to be considered highly temperature-dependent, and although there is evidence for recovery at very low temperatures from flow stress curves[7] and from electron micrographs,[8] contributions at these low temperatures must be relatively small. As the temperature of deformation is raised, simultaneous recovery becomes increasingly important in altering the cold-worked properties and structure. This interpretation is consistent with the data for properties that have been presented.

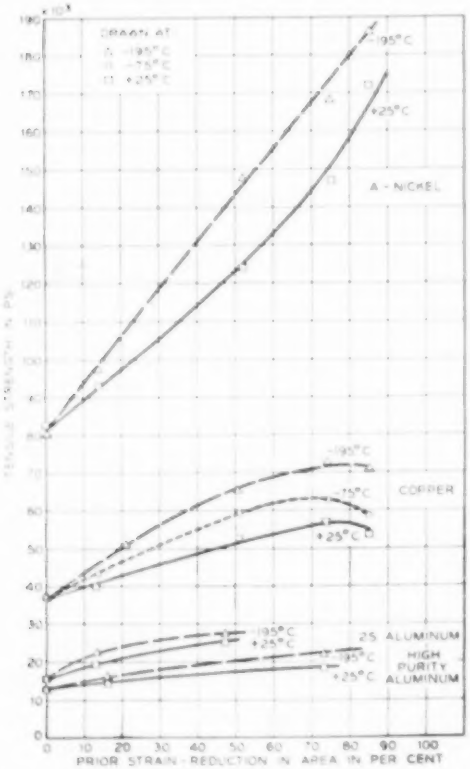

Fig. 3—Effect of temperature of drawing on room temperature tensile strength of nickel, copper, and aluminum.

Summary

As the temperature of deformation is lowered the flow strength and ultimate strength of a number of metals and alloys at room temperature are increased. Likewise, in materials in which lattice scattering predominates, electrical conductivity is decreased to a greater degree as the temperature of deformation is lowered. These effects are attributed to a decrease in simultaneous recovery during deformation as the temperature is lowered. Recovery is described as involving single atoms or small groups, and as highly temperature-dependent.

Data given for a number of commercial metals and alloys rolled at −195°C illustrate the increase in the strength properties that may be obtained by such treatment. The chief effects are on the properties determined largely by prior plastic treatment.

Acknowledgments

D. Wallace and R. J. Riley made important contributions in processing the materials; J. W. Nalencz conducted most of the measurements of mechanical properties; R. G. Treuting through helpful discussions has added substantially to the value of the paper. The encouragement and counsel of E. E. Schumacher in carrying out the work is gratefully appreciated.

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Deformation of Zinc Bicrystals by Thermal Ratcheting

by J. E. Burke and Anna M. Turkalo

IN 1923 Desch[1] pointed out that the grains in a metal which is anisotropic with respect to its thermal coefficient of expansion would contract differently upon cooling, and that the stresses developed might approximate the plastic strength of the metal. More recently Boas and Honeycombe[2,3] studied the behavior of several metals upon thermal cycling and observed that the stresses developed in anisotropic metals are great enough to produce slip lines in individual grains and a roughening of the specimen surface. This phenomenon they have named "thermal fatigue." The mechanism they propose involves essentially a kneading of the grains, the deformation being alternately in compression and tension in a given grain as the temperature is changed in one direction and then the other.

The present work was undertaken to investigate the possibility that an additional mechanism might operate to produce plastic deformation during thermal cycling—a "thermal ratchet" that depends upon a combination of grain boundary flow to relax the stress that develops between differently oriented grains upon raising the temperature and transcrystalline slip to relax the oppositely directed stress which develops on lowering the temperature. Thus, thermal cycling should produce a nonreversible distortion such that certain grains will change shape differently from their neighbors with a simultaneous displacement being produced at the grain boundary.

Temperature Dependence of Grain Boundary and Grain Strength

The critical resolved stress for the initiation of slip in metal grains is only mildly affected by temperature.[4] For example, in cadmium it decreases from 0.15 to about 0.05 kg per sq mm when the temperature is increased from 20° to 458°K and further temperature increase causes little further decrease.

On the other hand, the work of Kê[5] indicates that the grain boundaries behave in a viscous fashion that can be described[6] by the expression:

$$\tau = BV \exp(Q/RT) \qquad [1]$$

τ is the shearing stress on the boundary; B, a constant; V, the flow rate at the boundary; Q, the activation energy for grain boundary flow; R, the gas law's constant; and T, the absolute temperature. Eq 1 indicates that the stress necessary to cause a given grain boundary flow rate, V, decreases rapidly with increasing temperature. The value of the constant B is such that at sufficiently low temperature and ordinary strain rates deformation will occur preferentially by slip rather than by grain boundary flow. There is considerable evidence to indicate that extensive grain boundary flow can occur at elevated temperatures.[7]

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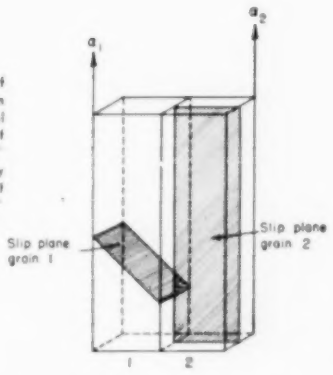

Fig. 1—Sketch of a bicrystal of an anisotropic metal with slip plane of grain 1 lying 45° to grain boundary and slip plane of grain 2 lying 90° to the boundary

Mechanism of the Ratchet

Consider the bicrystal shown in Fig. 1. In grain 1 the slip plane lies 45° to the boundary while in grain 2 the slip plane is 90° to the boundary. The coefficients of expansion of the grains in a direction parallel to the length of the crystal are a_1 and a_2, with $a_1 > a_2$ for the orientations shown. The sequence of events that can occur upon heating and cooling this specimen is illustrated schematically in Fig. 2.

Initially there is assumed to be no stress in the specimen (A). Upon heating, grain 1 attempts to become longer than grain 2, but is constrained by grain 2. Thus grain 1 is loaded in compression and grain 2 is loaded in tension, and a shearing stress is present across the boundary (B). As the temperature is increased, the stress will build up, and finally grain 1 will be plastically deformed by slip, since the greater stress is resolved on its slip planes. Any further heating will result in more slip and the stress will remain constant until some temperature T^* is reached where the stress can be relaxed by grain boundary flow.† At this relaxation temperature (C) a step will appear between grain 1 and grain 2. Further heating above T^* will cause grain 1 to become relatively longer, but no stress will appear because the grain boundary is too weak to support the stress (D). Upon cooling again, at T^* (E), the grain boundary will again be able to support a shearing stress, and upon cooling further, grain 1 will be loaded in tension and grain 2 in compression (F). When the decrease in temperature below T^* is sufficient to impose the critical shear stress upon the slip plane of grain 1, it will be stretched by slip.

† T^* is, of course, not a definite temperature but a temperature range where the grain boundary flow rate approximates the rate at which the grains would like to move relative to each other. It depends upon the difference between the coefficients of expansion, the heating rate, and the distance from the center of the bicrystal where the grains do not move relative to one another. Although this is a temperature range, for simplicity we shall consider it to be a definite temperature, T^*.

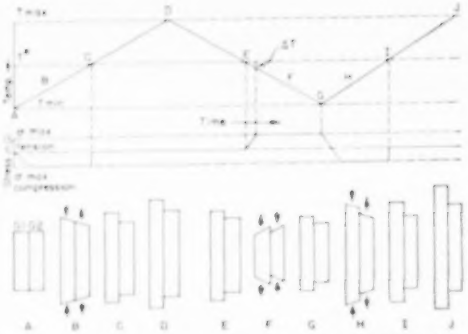

Fig. 2—Changes occurring during thermal cycling of a bicrystal of an anisotropic metal. Orientation of slip planes is the same as in Fig. 1.

Further cooling will produce additional elongation of this grain, while the stress remains essentially constant. Upon reheating, the stress will pass through zero (G) and then to compression to load the grains in the opposite direction (H, I). It should be noted that if a temperature decrease of ΔT below T^* is necessary to start slip, then a temperature decrease of $2\Delta T$ is necessary if the maximum stress is to be present when the specimen is reheated to T^*. If the specimen is cooled $2\Delta T$ below T^*, then the relaxation upon heating again to T^* will give an offset corresponding to the difference in expansion of the two grains over a temperature range ΔT. If the specimen is cooled more than $2\Delta T$ below T^*, the additional tensile strain will be recovered as compressive strain on reheating to T^*, and no greater displacement will occur.

Displacement per Thermal Cycle

The maximum amount of displacement that can occur per cycle is equal to the displacement that appears between grains when relaxation occurs at the grain boundary on heating. The amount the grain in compression elongates elastically when relaxation occurs is given by:

$$\Delta l = L \frac{S}{E} \qquad [2]$$

where L is the distance from the neutral center of the crystal; S, the compressive stress on the grain; and E, Young's modulus. The grain in tension will contract by approximately an equal amount so the total offset is given by:

$$2\Delta l = \Delta L = \frac{2LS}{E_{av}} \qquad [3]$$

where E_{av} is the average of Young's modulus for the two grains.

If the grains are of equal size, the compressive stress in one will be equal to the tensile stress in the other, and the maximum value of the stress, S_{max}, will be controlled by the strength of the weaker crystal. For the bicrystals used in this work S_{max} can be approximately calculated from S_c, the critical resolved stress for slip, by the relationship:

$$S_{max} = S_c / \cos\lambda \sin\chi \qquad [4]$$

where λ and χ are the angles between the tension axis and the slip direction and slip plane respectively.

Direction of Movement

One of the grains of the couple will be oriented so that it will flow more readily than the other under a tensile or compressive stress. This grain may have either a greater or a smaller coefficient of expansion than the other grain. Upon cooling below T^*, the grain with the larger coefficient of expansion will be loaded in tension, the other in compression. The major part of the deformation will occur in the softer grain. If the softer grain has the higher coefficient of expansion, it will be stretched and will become longer with each cycle. The other grain will be essentially unchanged. If the softer grain has the smaller coefficient of expansion, it will be strained in compression and become shorter on each cycle. Again, the other one will be essentially unchanged.

From these arguments we may draw the following conclusions:

1—Thermal cycling of a bicrystal should produce a continuing deformation of one grain that increases with the number of cycles.

2—The amount of deformation should be controlled by the flow strength and the value of Young's modulus for the metal.

3—For deformation to occur:

a—The temperature limits over which cycling occurs must be such that grain boundary flow occurs at the higher temperature and crystallographic slip occurs at the lower temperature.

b—The temperature range must be at least twice

Fig. 3—Before cycling.

Fig. 4—After 18 cycles.

Figs. 3 and 4—Typical scribed lines (vertical) on surface of zinc bicrystals before and after thermal cycling between 100° and 300° C. Grain boundary is horizontal line. X280.

that necessary to produce slip in the softer of the two grains.

4—Deformation will be largely confined to the softer grain. If it has the higher coefficient of expansion, it will lengthen; if it has the lower coefficient of expansion, it will shorten.

Experimental Procedure

The above arguments were tested by the thermal cycling of bicrystals of zinc with various pairs of orientations, and the observation of the offset produced at the boundary as a function of orientation difference, number of thermal cycles, and cycling temperature. Zinc[1] was selected because it is easy to grow zinc bicrystals, there is only one slip system which simplifies the analysis, and the coefficient of thermal expansion is highly anisotropic (63.9×10^{-6} per °C parallel and 14.1×10^{-6} per °C perpendicular to the unique[2] axis).

[1] The zinc for these experiments was kindly supplied by D. C. Jillson of the New Jersey Zinc Co.

The bicrystals were grown in horizontal graphite molds similar to those used by Chalmers.[3] No attempt was made to control orientations by seeding; it was found that self-seeded crystals had random orientations so that almost any desired orientation pair could be obtained by selection among a number of bicrystals. Specimens for cycling tests were cut with a jeweler's hack saw and then very heavily electropolished in 25 pct water solution of potassium hydroxide to give pieces about 1x4x1/8 in. with the grain boundary longitudinal. Lines were scribed on the large surface approximately perpendicularly to the grain boundary at intervals of about 0.5 cm with a Knoop diamond. Most of the specimens were sealed in a hydrogen atmosphere in pyrex glass tubes for cycling, but a few were cycled down to liquid nitrogen temperature without protection. Over the lower temperature range, the specimens were cycled by manual immersion first in liquid nitrogen, then in water at 25°C. Automatically lowering the specimen into a furnace and holding there until the specimen reached 300° and then removing it until the temperature had dropped to 100°C accomplished thermal cycling in the upper range. The low temperature cycles required about 5 min each, the high temperature cycles about 15 min.

Results of Experiments

Figs. 3 through 6 show examples of the displacements at the grain boundary on the polished surfaces of two zinc bicrystals. The scribed line is vertical and the grain boundary horizontal in each case. Fig. 3 shows the original condition, and Fig. 4 shows the displacement produced by 18 thermal cycles between 100° and 300°C. The offset at the grain boundary is about 0.01 mm on the specimen. Faint slip lines can be seen in the lower grain which was the softer one of the couple and in this case suffered all of the plastic deformation. The grain boundary migrated extensively during the cycling, and the dark areas on either side of the scribed line are surface roughening produced by thermal cycling with the grain boundaries in various positions. Figs. 5 and 6 show the displacement in another zinc bicrystal after 100 and 500 thermal cycles, respectively. The offsets are 0.05 and 0.14 mm. Both grains of the bicrystal were deformed by slip, but slip lines are more prominent in the softer grain (the lower grain in the photograph). No grain boundary migration was evident in this specimen. It is clear from these photographs that cycling between 100° and 300°C produced a relative displacement of one grain with respect to the other and the displacement increased with the number of cycles.

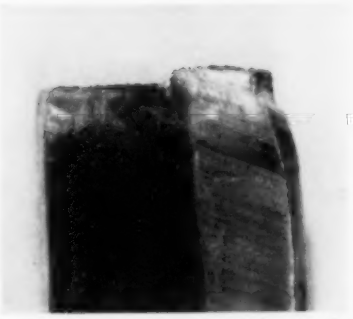

Fig. 7—End of zinc bicrystal after 500 thermal cycles between 100° and 300°C. X2

Cycling frequently caused the end of one grain to jut out beyond the other as shown in Fig. 7. This photograph also shows that slip lines are largely confined to one grain of the couple.

Thermal cycling between 25° and −180°C produced practically no offset at the grain boundary as the temperatures were too low for grain boundary relaxation. Figs. 8 through 11 show specimens before and after 100 cycles at the low temperature (Figs. 8 and 9) and high temperature (Figs. 10 and 11)

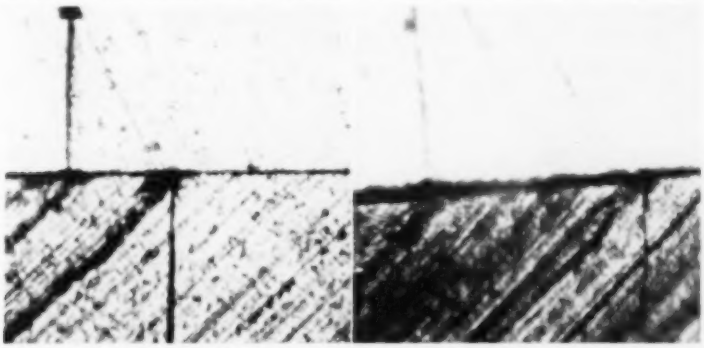

Fig. 5—After 100 cycles.

Fig. 6—Same line as that in Fig. 5 after 500 cycles.

Figs. 5 and 6—Typical scribed lines (vertical) on surface of zinc bicrystals after thermal cycling between 100° and 300°C. Grain boundary is horizontal line. X280

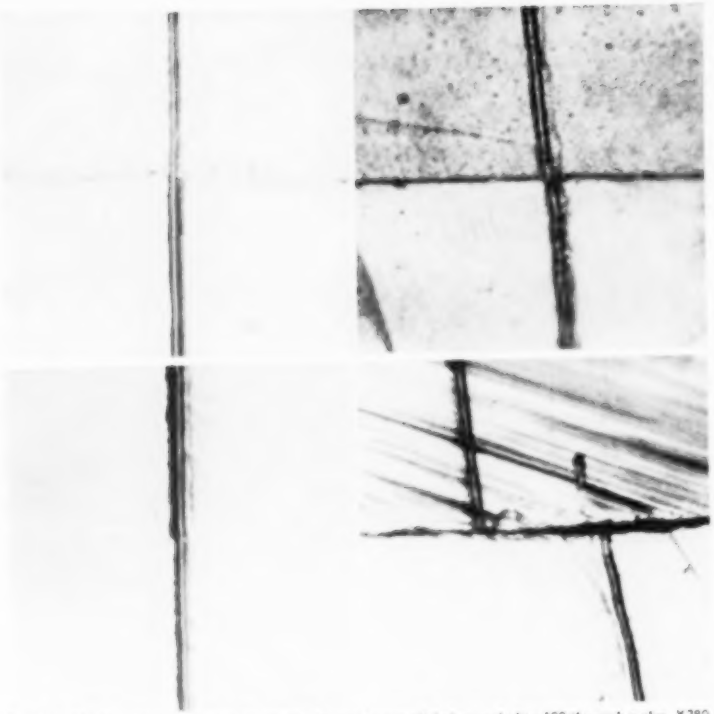

Fig. 8—Before cycling at the low temperature range (25° to −180° C).

Fig. 9—After 100 cycles at the low temperature range (25° to −180° C).

Fig. 10—Before cycling at the high temperature range (100° to −300° C).

Fig. 11—After 100 cycles at the high temperature range (100° to −300° C).

Figs. 8 to 11—Typical scribed lines on surface of zinc bicrystals before and after 100 thermal cycles. X280

range. Both specimens were cut from the same bicrystal and thus contained the same pair of orientations. The specimen cycled at the lower temperature range showed, in addition to the usual deformation by slip, considerable twinning near the grain boundary and also several cleavage cracks in both grains. Two such cleavage cracks in one of the grains are shown in Fig. 12 along with a twin extending from the end of one crack into the other grain. The specimen cycled in the higher range showed crystalline deformation exclusively by slip, which, as usual, was much more profuse in the softer grain of the couple, the upper grain of Fig. 11. There were no cleavage cracks in specimens cycled in the higher temperature range.

The observed displacements in the various specimens are summarized in Table I. The calculated values presented were obtained from eq 3 taking S_{ss} as 0.1 kg per sq mm and E_{ss} as 8000 kg per sq mm. In each case the calculated offset was higher than that actually observed. The discrepancy may be attributed to uncertainties in the selection of the value for S_{ss} and to variables which were not considered in the theory. For example, the grains often stuck at some points along the boundary and did not always show uniform movement over all of their length as predicted by the theory. Instead of there being one neutral position at the center of the sample length where no displacement occurred, there were frequently several such points. The behavior of specimen 2, which acted as if it had broken up into three units, is illustrated schematically in Fig. 13.

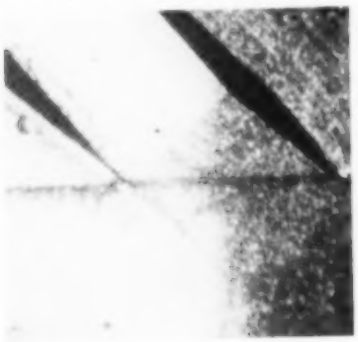

Fig. 12—Cleavage cracks in zinc bicrystal after 100 thermal cycles between 25° and −180° C. X100

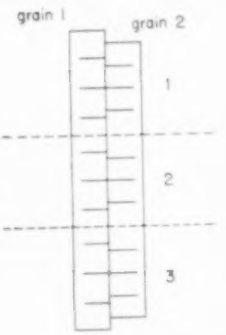

Fig. 13—Schematic drawing showing behavior of specimen No. 2 as three separate units during thermal cycling between 100° and −300° C.

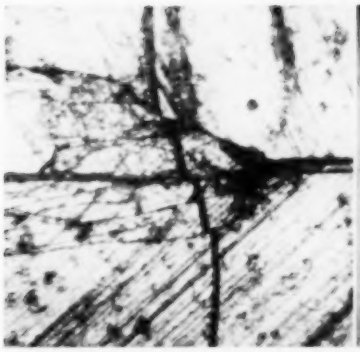

Fig. 14—Offsets of scribed line at different positions of migrating grain boundary in zinc bicrystal after 100 thermal cycles between 100° and 300°C. X280

Fig. 15—Grain boundary movement in a zinc bicrystal after 500 thermal cycles between 100° and 300°C. X7

Obviously the value selected for L (one half of the total specimen length) for the calculations of the maximum offset is too large for the actual effective L in this case. It seems probable that this "stickiness" at several points along the grain boundary was due to inclusions at the boundary, but no definite evidence was obtained for this.

In the samples cycled at the higher temperature the grain boundary migrated during the cycling to become highly irregular. Fig. 14 shows that this irregularity did not seriously hinder grain boundary flow and consequent displacement of the scribed line at the new positions of the boundary. However, it may have caused the decrease in the amount of displacement with increasing number of cycles which evidently occurred in specimen 2.

The softer grain had the higher coefficient of expansion in specimens No. 1, 3, and 5a, and as predicted, the softer grain increased in length. In specimen 2, the soft crystal had the lower coefficient of expansion, thus it shortened.

Several other observations made during this investigation deserve comment. In addition to grain boundary migration during the high temperature cycling, some recrystallization or polygonization occurred in the region immediately adjacent to the boundary in several of the samples. This can be seen in Fig. 15 where grains of slightly different orientations are visible along the grain boundary in the grain showing the least deformation. This specimen had been cycled 500 times between 100° and 300°C. Fig. 15 also shows that the offset was not always confined to a movement parallel to the longitudinal axis; movement occurred also in the thickness direction of the specimen, as would be expected. Apparently, as is evident from the "cliffs" at the boundary, grain boundary flow could occur reasonably easily at each position of the boundary, even when the grain boundary was rough.

It is of interest to note in the various photographs that the slip traces extend from the very grain boundary across the grain. This is to be expected since in the experiments described here, the stress arises at the grain boundary and initiates slip there—a situation quite different from the case in which an external stress is applied mechanically.[10, 11] Then the stronger grain can inhibit slip at the grain boundary in the softer grain. Twinning and/or bend plane action described by Jillson[12] as accommodation bending is not to be expected near the grain boundary in these experiments for the same reasons.

From these experiments it may be concluded that a ratchet mechanism does operate to produce a continuing distortion during thermal cycling of anisotropic metals within a sufficiently high temperature range. If the upper temperature limit is not high enough to permit grain boundary flow during cycling, the ratchet mechanism is lacking and only general crystalline distortion occurs. These experiments also indicate the need for further study and understanding of grain boundary behavior and stickiness before a complete picture of the ratchet is available.

Table I. Summary of Observed Displacements

Specimen No.	Temperature Range of Cycling, °C	No. of Cycles	Orientations* Grain a γ	Grain a λ	Grain b γ	Grain b λ	Calculated Maximum Offset, mm	Maximum Offset, mm
1	100-300	18	79°	80°	8°	16°	±0.01	0.11
2	100-300	100	42°	47°	60°	80°	±0.06	0.24
		500					0.14	1.05
3	100-300	500	72°	72°	0°	18°	~±0.5	1.88
4	25-180	50	39°	42°	54°	57°	None	
5a†	100-300	100	38°	45°	18°	32°	±0.08	0.20
			18°	32°	9°	9°	±0.13	0.40
5b†	25-180	100	38°	45°	18°	32°	None	
			18°	32°	9°	9°		

*Grain a—the softer grain of the couple.
Grain b—the harder grain of the couple.
γ—angle between specimen axis and slip plane (0001).
λ—angle between specimen axis and slip direction $\langle 11\bar{2}0 \rangle$.
† Specimens 5a and b were triercrystals cut from the same tricrystal.

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Technical Note

Melting of Undoped Silicon Ingots

by J. Hino and H. E. Stauss

INTEREST in silicon has arisen again in the past decade as a result of improvements in crystal rectifiers.[1] Although the preparation of silicon was first reported by Berzelius in 1880, the early product was of relatively low purity, and only the need for rectifiers in World War II led to the production of a 99.9+ pct pure powder. This material in crystalline form was consolidated into massive silicon for use, and the method developed was to melt it with selected added constituents as "doping" agents. Melting techniques, therefore, are of great importance.

There are two basic problems in producing silicon ingots free of doping additions; one is the prevention of spitting and the other is prevention of cracking of the ingot during freezing. The most satisfactory arrangement yet developed for producing massive silicon is to melt and freeze in a cylindrical quartz crucible surrounded by a concentric heating element and eccentric radiation shields or insulation. For example, use can be made of a tubular heater with a high frequency generator as the source of power and reflecting shields of alundum cylinders.

The spitting of silicon is related to gas evolution, and the gas comes from two primary causes—adsorbed gas and the reaction products of silicon and the crucible. Gas is also released from bubbles contained in the quartz crucible walls. Improved removal of adsorbed gas can be achieved by means of controlled melting and freezing. The seriousness of the problem in vacuo is reduced with an electrically operated mechanical movement of the high frequency power coil. The upper portion of the powder charge is melted first and the high frequency coil lowered until the powder is completely molten. During cooling the high frequency coil is raised slowly. These means also reduce the final nonviolent extrusion of large beads of metal through the ingot top during freezing. Better control of spitting and bead extrusion is obtained when melting is done under helium at atmospheric pressure instead of in vacuo.

The problem of reaction between silicon charge and crucible in practice is confined to the reaction between silicon and quartz. This[2] apparently is:

$$Si + SiO_2 \rightleftharpoons 2SiO$$

The part that this reaction plays in spitting has not been isolated for separate study. SiO is a volatile vapor at the melting point of silicon and is released freely during melting in vacuo, but hardly at all in helium at atmospheric pressure.

The cracking of ingots is a major difficulty in melting silicon, and its prevention requires special melting techniques or the addition of "toughening" agents such as aluminum or beryllium.[3] The cracking of the ingots has been explained as being the result of the expansion that occurs upon freezing,[4] although direct observation of freezing ingots reveals visible cracks on the surface only after a red heat has been reached, suggesting that cracking is the result of differential contraction of silicon and quartz. Silicon wets quartz, and the ingot adheres tightly to the crucible. Therefore as ingot and crucible cool, the two either have to pull apart, or at least one must crack. Surprisingly, in spite of the relative thinness of the quartz and the thickness of the ingot, the ingot and the crucible both crack. Microscopic and X-ray[5] studies fail to show any plastic flow other than twinning in the ingots. Slow cooling fails to prevent cracking. Another possible solution to cracking is to weaken the crucible. Use of thin-walled crucibles finally led to success with fused quartz crucibles with a wall thickness of 0.25 to 0.50 mm. With such thin-walled fused quartz crucibles consistently uniform success is secured in producing sound ingots 30 mm in diam from the purest available grade of silicon (99.9+) without the use of any type of addition. Melts are made in the size range of 50 to 100 g.

Omission of a deliberately added doping agent is not sufficient to insure pure ingots. The reaction of silicon with crucibles and the resultant solution of impurities in the silicon is well-established.[6] In this laboratory, the presence of Al, Be, and Zr has been found spectroscopically in ingots melted in contact with alumina, beryllia, and zircon. The best crucible materials reported in the literature are MgO and SiO_2. Use of MgO in this laboratory has resulted in a heavy deposit of magnesium on the furnace walls, showing that a reduction of the magnesia occurred and the resulting magnesium removed from the melt by volatilization. In the case of quartz, the silica is reduced and SiO liberated to deposit on the equipment walls. There probably is real danger that oxygen is dissolved in the ingot when either magnesia or silica is used as the crucible material. Preliminary analyses by Dean Walter in his vacuum unit in this laboratory[7] indicate the presence of oxygen in undoped silicon melted in quartz.

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Surface Areas of Metals and Metal Compounds: A Rapid Method of Determination

by C. Orr, Jr., H. G. Blocker, and Susan L. Craig

WITHIN recent years gas adsorption methods have been developed for measuring the surface area of finely divided materials and have become extremely valuable in research on the corrosion and the catalytic activity of metals. Rather elaborate apparatus is required, and a single determination is so time-consuming that these methods have not been utilized to the fullest extent; the methods are unsuited for most routine control work such as that encountered in powder metallurgical operations and in processes employing metal catalysts. These difficulties are largely eliminated, and surface area is reduced to a routine determination if the liquid-phase adsorption of a surface-active agent such as a fatty acid can be used. When the affinity of the fatty acid carboxyl group for the solid surface is greater than its affinity for the solvent, a unimolecular layer of orientated fatty acid molecules will be formed at the solid-liquid interface in a manner similar to that of a compressed fatty acid film on a water surface. The measurement of surface area is then reduced to a measurement of fatty acid adsorption.

This propitious circumstance, first investigated by Harkins and Gans,[1] has been employed with somewhat inconclusive results by a number of investigators in evaluating the surface properties of metals, metal catalysts, and metal oxides. The specific surface area values for nickel and platinum catalysts, determined from the adsorption of a number of fatty acids from various solvents, were found by Smith and Fuzek[2] to agree with values calculated by the gas adsorption technique of Brunauer, Emmett, and Teller,[3] the so-called BET technique. And recently Orr and Bankston[4] have also reported good agreement between nitrogen gas and stearic acid adsorption results in the measurement of the surface areas of clay materials. On the other hand, Ries, Johnson, and Melik[5] found only order-of-magnitude agreement between these two methods in studying supported, cobalt catalysts having specific surface areas as great as 420 sq m per g; the reason is partially attributable to the very porous nature of the materials. Greenhill,[6] investigating the adsorption of long-chain, polar compounds in organic solvents on a number of metal powders, concluded that a unimolecular layer of stearic acid was formed on exposure of the solid to the acid solution and that the presence of an oxide or another film did not alter this result. Furthermore, the adsorption process appeared to be the same whether or not the sample was degassed prior to exposure to the solution. Greenhill estimated the surface area of one of the powders he investigated from microscopic diameter measurements, and obtained a rough check with surface area evaluation. Russell and Cochran[7] found moderate agreement for alumina surface area results by fatty acid and gas adsorption methods. In addition, they also found that the prolonged heating and evacuating pretreatments previously used by investigators were unnecessary. The present work, however, considerably extends these previous investigations, shows that fatty acid adsorption can be used to determine the surface area of a variety of metals and metal compounds, offers further confirmation of the correctness of gas adsorption methods, and presents a simplified technique for the determination of the metal surface area which is suitable for routine work.

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Experimental Technique

Basically, the fatty acid adsorption method is quite simple. It consists of exposing a sample of the material of which the surface area is desired to a fatty acid solution of known concentration. By analysis of an aliquot of the solution, the concentration after adsorption has occurred may be determined. The difference between the initial quantity of acid in solution and the final quantity is that quantity of acid adsorbed by the sample. The specific surface area of the adsorbent material may be calculated from the quantity adsorbed and the weight of the sample.

In agreement with the findings of others as outlined above, it was found entirely unnecessary to degas or pretreat the nonporous materials employed other than by drying them thoroughly. However, precaution was necessary so that the dried sample entered the fatty acid solution with little exposure to moisture.

The effect of moisture on the interaction of stearic acid with finely divided materials has been thoroughly investigated by Hirst and Lancaster.[8] They found the presence of water merely reduced the amount of acid adsorbed by powders such as TiO_2, SiO_2, TiC, and SiC. With reactive materials such as Cu, Cu_2O, CuO, Zn, and ZnO, however, water was found to initiate chemical reaction. Only with ZnO was reaction observed when the solid and the solu-

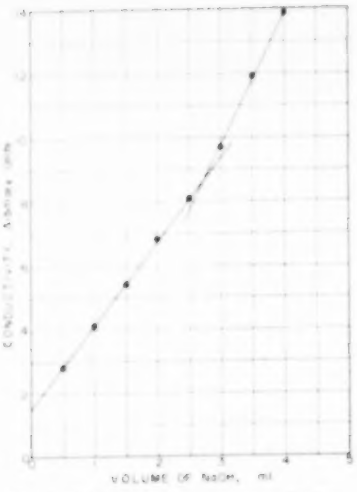

Fig. 1—Conductometric titration curve for iron (see sample calculation).

tion were carefully dried. The investigators believe that sufficiently dry conditions were not attained in this case.

Methanol was employed as the solvent in all the work reported here, although hexane and benzene have been used with equally good results. Chemically pure methanol, further purified by distillation (the fraction boiling between 64° and 65.5°C was retained) then dried by storage over calcium sulphate, was used together with the best grade of stearic acid (melting point, 68° to 69.5°C) from the Eastman Organic Chemicals Co.

The solid material and the stearic acid-methanol solution were vigorously shaken together for at least 30 min. Materials with large surface areas require somewhat more time. Despite indications that adsorption is not absolutely complete even after several hours, it is so nearly completed in a few minutes that much longer periods of agitation are deemed unwarranted. Moreover, if a reaction between the stearic acid and the metal should occur because of the presence of traces of water, additional justification is offered for as short a contact period as possible.

The method hinges on the determination of fatty acid in solution, and several methods are available. Smith and Fuzek[7] evaporated an aliquot of solution to dryness and weighed the residue. Colorimetric methods have been described by several investigators.[8-10] Hutchinson[11] and Greenhill[7] employed a surface balance. Saunders,[12] with water-soluble fatty acids, used a surface tension balance. The conductometric titration method of Maron, Ulevitch, and Elder[13] was chosen for this work because it was quite rapid, simple, and accurate; also the necessary equipment was readily available. However, any of the above methods could have been used.

In our technique, after adsorption was considered complete, an aliquot of the equilibrated solution was centrifuged, when necessary, to remove suspended matter. Additional pure methanol was added to make sufficient solution to cover the electrodes, and this solution was titrated conductometrically with sodium hydroxide. The milliliters of NaOH added to obtain the end point is that quantity of NaOH required to saponify the fatty acid and to satisfy the blank requirements of the alcohol. One molecule of NaOH will saponify one molecule of fatty acid; and one molecule of a straight chain fatty acid, regardless of the length of the chain, occupies, upon adsorption, an area of 20.5A², or 20.5×10^{-20} sq m.[14] As discussed by Maron et al.,[13] the blank requirement of the alcohol is an unalterable characteristic of each batch of alcohol. Its value may be determined by separate experiment; allowance for its value must be made in each calculation. Sample calculations for iron powder are given below. The corresponding conductometric titration results are plotted in Fig. 1.

Data:

Iron sample weight	16.8779 g
Pure methanol blank requirements	0.007 ml NaOH per ml
Initial stearic acid-methanol solution concentration (approximately 0.8 saturated)	0.6478 g per liter
Volume of initial stearic acid-methanol solution added to sample	100.00 ml
Volume of aliquot of final stearic acid-methanol solution removed from sample	25.00 ml
Volume of pure methanol added to aliquot	100.00 ml
Normality of NaOH solution	0.0191 N
Volume of NaOH to titrate final aliquot	2.75 ml

Calculations:

NaOH to saponify stearic acid in aliquot:
$2.75 - (100.00 + 25.00)0.0070 = 1.87$ ml

Stearic acid present before adsorption:
$100/1000 \times 0.6478 \times 1/284.5 = 22.77 \times 10^{-5}$ mols

Stearic acid remaining after adsorption:
$100/25 \times 1.87 \times 1.91 \times 10^{-5} = 14.29 \times 10^{-5}$ mols

Stearic acid adsorbed:
$(22.77 - 14.29) \times 10^{-5} = 8.48 \times 10^{-5}$ mols

Specific surface area of iron powder:
$8.48 \times 10^{-5}/16.8779 \times 6.023 \times 10^{23} \times 20.5 \times 10^{-20} = 0.62$ sq m per g

While no definite rule can be given, best results have been obtained when the weight of sample was adjusted so that approximately one half of the acid was adsorbed from the solution. If too little sample was present, the quantity removed by adsorption was small and could not be accurately determined, while, if too much sample was present, the sample surface was not completely covered.

Specific surface area measurements were also obtained from the adsorption of nitrogen gas at the temperature of liquid nitrogen by the procedure described by Emmett[15] and by Emmett and Brunauer,[16-18] and with ethane gas at the temperature of liquid oxygen by the procedure of Wooten and Brown[19] as modified by Lauterbaugh, Laskin, and Leach.[20] In the case of nitrogen adsorption, samples were evacuated at 250°C for at least 18 hr to a pressure of less than 5×10^{-3} mm Hg before adsorption was started. Samples having a total surface area of at least 5 sq m were used in every case. With ethane adsorption, evacuation at 250°C was continued—sometimes for 48 hr—until a pressure of at least 10^{-4} mm Hg was attained before beginning adsorption.

The surface areas of some of the materials studied were also estimated from particle diameter measurements made directly with an optical microscope fitted with an optical micrometer, or, in the case of the finer powders, were made from electron micrographs of the powders. From 100 to 200 particle diameters were measured in every case, and median particle diameters were determined by plotting the measured diameters on log-probability paper as described by DallaValle.[8] Their surface area was then calculated assuming that all the particles were spheres of the determined median diameter. All specific surface area data are tabulated in Table I. With the exception of copper, which was approximately spherical, all of the powders employed consisted of irregularly shaped particles. In addition, two porous materials, a nickel catalyst and an adsorbent alumina, were included.

Discussion of Results

Livingston,[9] by a critical survey of published adsorption data, established a value of 15.4Å² as the cross-sectional area of an adsorbed nitrogen molecule, an area believed nearly correct for most adsorbents. In the same article a value of 22.5Å² is given for the area occupied by an adsorbed ethane molecule. These values were used in calculating gas adsorption results. The area occupied by an adsorbed straight-chain fatty acid molecule on a liquid surface has been established to be very nearly 20.5Å². This value, generally accepted also for the area occupied on a solid surface,[4, 10] was used in this work. General agreement for nonporous materials is to be expected between the surface area obtained by microscopic measurement and the other methods. For porous materials such as the high surface nickel and adsorbent alumina, no agreement can be expected.

Table I. Comparison of Surface Area Measurements

Metal or Compound	Condition of Preparation, Pretreatment, Etc.	Surface Area (Sq m per g) Nitrogen Gas	Ethane Gas	Stearic Acid	Microscopic
Iron	Medicinal iron reduced with H_2	0.61		0.62 0.66	0.43 0.82*
Nickel		0.18	0.23 0.19	0.23 0.24 0.19	
Nickel	Reduced from the hydroxide with H_2 and deactivated with C_2H_2	23.7		24.50 24.20	0.35
Zinc		0.38		0.43	
Alumina	Prepared for use as an abrasive	2.18	2.33	2.34 2.11	1.00
Alumina	Prepared for use as an adsorbent	195.2		181.2 196.6 166.8 171.9	
Cadmium		0.12		0.17	
Tungsten carbide†	Pure tungsten powder and lamp black heated to 1400° C in an induction furnace	0.11		0.11	0.05
Tantalum carbide†	Pure tantalum metal and carbon heated to 2300° C with metallic aluminum and purified mechanically and chemically	0.09		0.09	0.07
Copper	100 mesh	0.23		0.23	0.24 0.23
Copper	200 mesh	0.38		0.34	0.37 0.26*

* From electron micrograph.
† Obtained from Kennametal, Inc., Latrobe, Pa.

All things considered, the results of this investigation give added confirmation of the validity of the gas adsorption methods as a means of measuring surface area and indicate that fatty acid adsorption is a valuable companion technique. Liquid-phase adsorption, because extensive sample evacuation or heating is unnecessary, and because the method is rapid, should be usable with heat-sensitive and high vapor pressure materials as well as in process control work with metals and with other more stable materials.

Acknowledgment

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Technical Note

Crystallographic Angles for Titanium and Zirconium

by Carl J. McHargue

THE angles between the crystallographic planes in cubic crystals have been given by Bozorth,[1] in magnesium, zinc, and cadmium by Salkovitz,[2] and in tin by Nicholas.[3] The determination of the orientation of single crystals and the study of plastic deformation require the knowledge of these angles and are often facilitated by use of a standard projection. Tables of these angles and standard projections have not been published for titanium or zirconium which have c/a ratios considerably less than those hexagonal metals for which this data is available. Accordingly, Table I has been prepared giving these angles. Fig. 1 presents a standard (0001) projection of titanium.

The angle between $(h_1k_1i_1l_1)$ and $(h_2k_2i_2l_2)$ was calculated by means of the formula:

$$\cos\phi = \frac{h_1h_2 + k_1k_2 + \frac{1}{2}(h_1k_2 + h_2k_1) + \frac{3}{4}\frac{a^2}{c^2}l_1l_2}{\left[(h_1^2 + k_1^2 + h_1k_1 + \frac{3}{4}\frac{a^2}{c^2}l_1^2)(h_2^2 + k_2^2 + h_2k_2 + \frac{3}{4}\frac{a^2}{c^2}l_2^2)\right]^{\frac{1}{2}}}$$

The c/a ratio for titanium is taken from the data of Clark[4] and that for zirconium from data given the Metals Handbook.[5] For all practical purposes Fig. 1 serves for zirconium as well as titanium, as can be seen from the similarity of the values of all angles.

Table I. Angles between the Crystallographic Planes in Close-Packed Hexagonal Titanium and Zirconium

$(h_1k_1i_1l_1)$	$(h_2k_2i_2l_2)$	Titanium, c/a = 1.5873	Zirconium, c/a = 1.5803
0001	$10\bar{1}0$	12° 54′	12° 55′
	$10\bar{1}1$	14 41	14 42
	$10\bar{1}6$	16 56	16 58
	$10\bar{1}5$	20 05	20 31
	$10\bar{1}4$	24 37	24 39
	$20\bar{2}7$	25 27	27 41
	$10\bar{1}3$	31 23	31 28
	$20\bar{2}5$	36 15	36 17
	$10\bar{1}2$	42 31	42 32
	$20\bar{2}3$	50 42	50 45
	$10\bar{1}1$	61 23	61 25
	$20\bar{2}1$	74 44	74 47
	$10\bar{1}0$	90 00	90 00
	$21\bar{3}2$	67 35	67 37
	$21\bar{3}1$	78 21	78 22
	$21\bar{3}0$	90 00	90 00
	$11\bar{2}8$	21 36	21 40
	$11\bar{2}6$	27 53	27 55
	$11\bar{2}4$	38 26	38 28
	$11\bar{2}2$	57 47	57 50
	$11\bar{2}0$	90 00	90 00
	$12\bar{3}2$	67 35	67 37
	$12\bar{3}1$	78 21	78 22
	$12\bar{3}0$	90 00	90 00
1010	$21\bar{3}0$	19 06	19 00
	$11\bar{2}0$	30 00	30 00
	$12\bar{3}0$	40 54	40 54
	$01\bar{1}0$	60 00	60 00

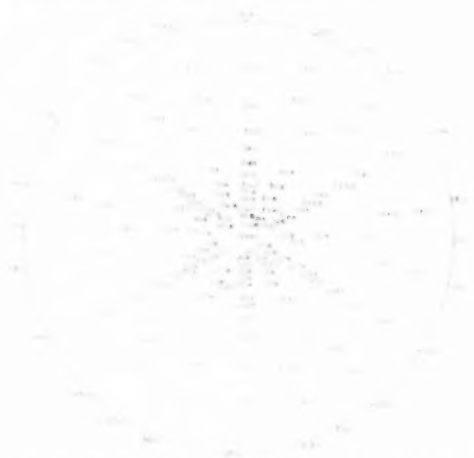

Fig. 1—Standard (0001) projection for titanium, c/a = 1.5873.

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Effect of Applied Stress on the Martensitic Transformation

by S. A. Kulin, Morris Cohen, and B. L. Averbach

The martensitic transformation can be initiated by elastic stresses at temperatures above M_s in a steel containing 20 pct Ni and 0.5 pct C. Shear strains and normal tensile strains acting on a potential habit plane promote the transformation but compressive strains oppose it.

THE strain sensitivity of the martensitic transformation has long been recognized. It is now well known that martensite formation can be induced by plastic deformation at temperatures below, and not too far above, the M_s point.[1] However, the exact role of elastic vs. plastic strains and of the concomitant stresses has proved to be a very elusive matter.

Many investigators[2-6] of the steel hardening reaction have made use of the stresses resulting from volume changes to account for such observations as the existence of retained austenite and its variation across the section of a bar, the change in the amount of retained austenite with quenching rate, and the self-stopping nature of the transformation when the cooling is stopped. The effect of applied strain as an independent variable has been studied in 70 pct Fe-30 pct Ni alloys by Scheil[7] and McReynolds.[8] Some data are also available for lithium and Li-Mg alloys,[9] Cu-Zn,[10] Cu-Sn and Cu-Al alloys,[11] and austenitic stainless steels.[12-15]

It has been found that martensite formation can be induced isothermally, even at temperatures above M_s, by mechanical deformation of the parent phase. The competitive nature of plastic yielding by slip and by the martensitic reaction has been depicted by Scheil.[7] Basically, Scheil postulated that: 1—at temperatures not too far above M_s (where austenite is less stable than martensite), a critical resolved shear stress (within the elastic range) is required to promote the transformation to martensite; 2—at and below M_s, the austenite lattice becomes mechanically, as well as thermodynamically, unstable and shears over spontaneously into martensite without the application of external stress; and 3—at temperatures sufficiently far above M_s, the critical resolved shear stress for martensite formation increases to a level above that required for slip, and plastic flow then supersedes the transformation when external stress is applied.

Scheil's concepts were tested by McReynolds[8] who found no change in the elastic moduli (measured both statically and dynamically) of 71 pct Fe-29 pct Ni alloys in the vicinity of the M_s temperature. Since the moduli did not approach zero or become negative on cooling to M_s, it was concluded that the austenite lattice does not become mechanically unstable at M_s, as postulated by Scheil. McReynolds also reported that M_s is not raised by elastic stresses. Accordingly, plastic deformation was considered to stimulate the transformation by generating martensite nuclei in the distorted regions of the parent phase.

The above issues with regard to the role of applied stress require clarification, if a basic understanding of the martensitic transformation is to be obtained. For example, in the reaction-path theory,[16,17] it is postulated that strain embryos exist in the austenite that provide part of the activation energy for the nucleation process. These embryos are visualized to be regions of localized strain in which the atoms in the austenite lattice are displaced part way along the path to their ultimate positions in the martensite. Consequently, it would

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Fig. 1—Stress vs. plastic strain at temperatures above M_s. Horizontal discontinuities indicate stress induced martensitic transformation. 20 pct Ni 0.5 pct C steel.

be predicted that applied stress, even within the elastic range, might contribute to the displacement in the embryos and thus enable them to achieve the critical strain for nucleation at temperatures above M_s. Furthermore, because of the unique nature of the atomic displacements that convert the austenite lattice into martensite,[1] it can be expected that the direction, as well as the magnitude, of the applied stress should be an important variable in the nucleation process. This paper describes a number of critical experiments that were designed to resolve these questions.

Experimental Details

An alloy steel containing 20 pct Ni and 0.5 pct C was chosen for this investigation because its normal martensitic transformation occurs in a convenient subzero temperature range, and the extent of isothermal martensite formation[2-5] is negligibly small, thereby minimizing any possible complication from the time variable during the stress studies. The complete analysis was 19.99 pct Ni, 0.52 pct C, 0.37 pct Mn, 0.47 pct Si, 0.010 pct P, 0.015 pct S, 0.04 pct Cr, balance Fe.

The specimens were austenitized at 1095°C for 30 min in a purified nitrogen atmosphere, and quenched in oil at 20° to 22°C. They were then sealed in evacuated Vycor tubes and stress relieved at 480°C for 30 min, followed by furnace cooling to room temperature. The structures were completely austenitic after the foregoing cycle, the M_s temperature being about −37°C.*

Fig. 2—Stress vs. plastic strain at temperatures above M_s. 20 pct Ni 0.5 pct C steel. Compare with Fig. 1.

* In this alloy, the transformation starts with a burst,[6] and hence $M_s = M_b$. However, there are certain conditions (to be discussed later) under which $M_s > M_b$.

treatment was introduced to avoid the superimposition of quenching stresses on the applied stresses during subsequent cooling. This factor will be discussed later.

Prior to microexamination, the specimens were usually tempered at 315°C for 10 sec in order to "darken" any martensite that might exist.

Tensile stresses were applied to austenitized and stress-relieved specimens having a gage length of 2.5 in. and a gage diameter of 0.250 in. The loads were increased in small increments, with intermediate unloading. The permanent set, if any, was determined with two electrical resistance strain gages, mounted longitudinally on each specimen. For simultaneous transformation measurements, the changes in electrical resistance of the specimens were observed by means of a Kelvin double bridge, using potential leads spotwelded near the fillets. The loading was carried out at various temperature levels, maintained constant to ± 0.5°C. The subatmospheric temperatures were achieved by adding powdered CO_2 to the oil bath in which the specimens were immersed during the testing. Other types of stress experiments will be described later in the paper.

Martensitic Transformation Under Applied Tensile Stress

The foregoing "load-unload" procedure was applied to austenitized and stress-relieved specimens of the 20 pct Ni-0.5 pct C steel at temperatures between 20° and −25°C. In each instance, stress vs. plastic strain (permanent set) was plotted as shown in Figs. 1 and 2. The method was capable of detecting plastic strains down to $\pm 2 \times 10^{-4}$. Plastic strains up to 0.012 were studied here.

The sudden yielding denoted by the horizontal lines in Figs. 1 and 2 corresponds to martensite formation. This was established both by the accompanying decrease in electrical resistance and by subsequent microexamination. At temperatures in the vicinity of −15° to −25°C, sharp clicks could be heard as rather large amounts of martensite formed in bursts, even though these temperatures were definitely above the martensite range for the unstressed material.

No transformation was detected in the specimens strained at 20°C and there was only a slight trace at 0°C; in these cases, the electrical resistance increased progressively with the plastic deformation. However, at −15°C and below, appreciable quantities of martensite were produced during the tests, and (for a given permanent set) increased with decreasing temperature. This trend is illustrated by the data in Table I.

Even more significant is the fact that the inception of the transformation was induced at temperatures above M_s or M_b by tensile stresses well within the elastic range. For example, at 20°C the elastic limit (corresponding to a permanent set of 2×10^{-4}) was about 17,000 psi and it would be expected to be at least as high at −25°C. However, at the latter temperature, the transformation was triggered by a tensile stress of only 1000 psi. It may be concluded that the M_s (and M_b) temperature can be raised by elastic tensile stresses.

From the results of these experiments, the yielding by martensite formation and plastic flow by slip may be regarded as two competitive deformation mechanisms. While it is likely that either may affect the other, neither one is necessary for the other, and the martensitic transformation sets in before the slip process if the tensile stress is applied at temperatures not too far above M_s. The onset of the transformation due to tensile stresses within the elastic range results in a "premature" yielding, and therefore the material may have an abnormally low yield strength. However, it is evident from Fig. 2 that the formation of martensite under stress produces more effective "strain hardening" (cf. curves for −15° and −20°C) than does the ordinary slip mechanism (cf. curves for 20° to 0°C), and hence relatively high tensile strengths may be encountered despite the low yield strengths.

It was observed that the austenite twin boundaries are sometimes preferred regions for the nucleation of martensite under the application of stress. As shown in Fig. 3, the initial martensitic plates were nucleated in pairs, like chevrons, extending more or less symmetrically into both the twin and the mother crystal. This phenomenon is readily explained in terms of the transformation strain analysis of Machlin.[7] The cooperative atomic displacements involved in the formation of a martensite plate require considerable movement in the surrounding austenite, and the associated strain energy contributes materially to the energy of nucleation. However, since the twin boundary is a mirror image of the lattice on either side, the formation of a pair of plates reduces the strain energy requirements because both plates tend to move the intervening austenite in about the same direction. Under these conditions, the twin boundaries should be favored sites for nucleation, provided that the plates form cooperatively in pairs. In the specimen represented by Fig. 3, only a minute amount of transformation occurred at 0°C on plastically straining to 0.012 in. per in., but most of it took place along twin planes in the form of double plates. At the lower straining temperatures where substantial quantities of martensite appeared, the plates had a more conventional distribution.

Martensitic Transformation Under Bending Stress

Because of the shear-like nature of the martensitic reactions along the habit plane, it might be assumed that the stimulating effect of elastic tensile stress is caused solely by the resolved shear component of the applied stress. If this were the case, uniaxial compressive stress should produce similar results. A simple bending experiment will suffice to prove that such a shear criterion for the activation of the transformation by stress is invalid.

A $\frac{1}{2}$x$\frac{3}{8}$x4 in. bar of the 20 pct Ni-0.5 pct C steel was austenitized and stress relieved in the usual way. It was then loaded in uniform bending at 20°C to a maximum fiber stress of 14,000 psi, this being below the elastic limit of the material at room temperature. While loaded in this way, the specimen was slowly cooled until a burst of transformation occurred. The temperature at that instant was −21.9°C, which was well above the M_s = M_s for the unstressed material. The specimen was immediately heated to room temperature, unloaded, and tempered to "darken" the martensite.

Metallographic examination revealed (Fig. 4) that the transformation had taken place in the part of the specimen subjected to tensile stress, whereas no transformation was observed in the part under compressive stress. Also, the neutral axis of the bent bar had shifted toward the compressive side, presumably because of the yielding on the tensile side due to the transformation there.

Table I. Extent of Transformation as a Function of Tensile-Test Temperature

Temperature of Tensile Test, °C	Pct Martensite Formed (For Plastic Strain of Approximately 0.012)
20	0
0	Trace
−15	0.5
−20	12.3
−25	28.0

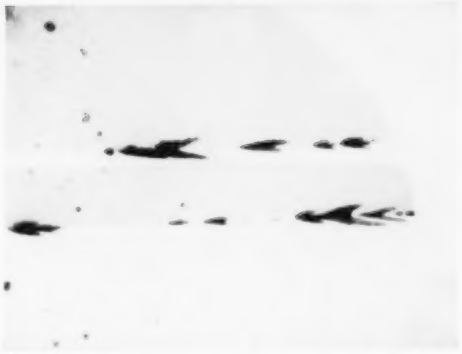


Fig. 3—Martensitic chevrons lying along twin boundaries of 20 pct Ni-0.5 pct C steel after plastically straining at 0°C. Tempered at 315°C for 10 sec. Etched with 4 pct nital and 1 pct zephiran chloride. X500.

Fig. 4—Longitudinal (vertical) section through bar of 20 pct Ni-0.5 pct C steel cooled to M_s temperature while subjected to elastic bending. Top is compression side, bottom is tension side. Tempered at 315°C for 10 sec. Etched with 4 pct nital and 1 pct zephiran chloride. X8.

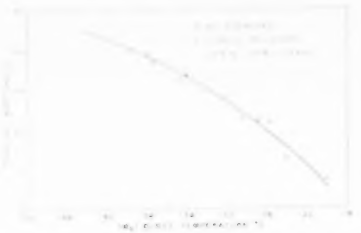


Fig. 5—Percent martensite formed during the burst vs M_s temperature.

These findings demonstrate not only that elastic tensile stresses promote the transformation, but also that elastic compressive stresses do not act in a corresponding manner. Inasmuch as the shear stress on geometrically similar planes is the same in both the tensile and compressive regions of the bent specimen, then it must be the sign of the normal stress that is responsible for the striking difference in transformation behavior. Thus, it is now clear that, although the component of shear stress along a habit plane undoubtedly plays an important part in the activation process, the component of stress normal to this plane is likewise important. By the same token, a critical resolved shear stress or shear strain or shear-strain energy cannot provide a suitable criterion for the initiation of the transformation, because the contributions of the magnitude and direction of the normal stresses are thereby neglected.

Effect of Residual Stresses on Transformation

It is evident from the results presented thus far that residual stresses may influence the transformation and, in fact, this was the reason for stress relieving the austenitic specimens prior to the applied stress studies. The role of residual stresses may be illustrated in the following way: Two series of $\frac{1}{4}$ in. diam x $\frac{1}{2}$ in. long specimens were austenitized, one series was then stress relieved at 480°C for 30 min and the other was not. On subsequent cooling, the stress-relieved specimens were found to have M_s temperatures between −34° and −40°C (average −37°C), while the specimens without stress relief had M_s temperatures between −20° and −30°C (average −26°C). Furthermore, the amount of transformation occurring during the burst decreased with increasing M_s temperature. This correlation was the same for both series of specimens as shown in Fig. 5.

In the nonstress-relieved series, it was found that the quantity of martensite formed during the burst varied markedly across the section. The specimen with the highest M_s at −20°C was entirely austenitic around the rim, the transformation being confined to the central portion. Fig. 6 shows the austenitic rim on a transverse section, with some of the martensite formed toward the interior of the specimen. The other samples in this series with lower M_s temperatures had progressively less of this rim effect, and the stress-relieved specimens contained a relatively uniform amount of transformation across the section, as in Fig. 7a.

These results are attributable directly to the residual stresses in the austenitized steel. The quenching from the austenitizing temperature puts the specimens into a state of tension in the interior and a state of compression near the surface. As in the case of the bending experiments, the transformation takes place "prematurely" in the regions sustaining tensile stresses, but not in those sustaining compressive stresses. The higher the residual tensile stress in the core of the specimen, the sooner does the burst occur on cooling and the more restricted it becomes. With little or no residual stress present, the M_s temperature is lower and the burst takes place more uniformly throughout the specimen.

On the basis of this reasoning, it may be predicted that the completely austenitic rim remaining after the burst in a nonstress-relieved specimen, such as that of Fig. 6, should undergo transformation on cooling below the M_s temperature, since the martensite formed in the core causes an expansion there which reduces the tensile stresses in the core as well as the compressive stresses in the rim. This was confirmed: on cooling some 10° to 15°C below M_s, the transformation became fairly uniform across the transverse section of the specimen.

To show the effect of the superimposition of residual and applied stresses, a $\frac{1}{4}$ in. diam x $2\frac{1}{2}$ in. rod was water quenched from the austenitizing temperature, placed in elastic bending along the central portion, and then cooled until a burst of transformation was noted by electrical resistance measurements. After tempering the specimen to "darken" the martensite, cross-sections were examined metallographically in regions subjected to the bending and in regions external to the bending clamp. Fig. 7b illustrates the latter situation, with a symmetrical core of transformation corresponding to the state of high tension in the interior produced by the water

Fig. 6—Martensite formation in 20 pct Ni 0.5 pct C steel austenitized at 1095°C, quenched to room temperature, and subcooled to M_s (−20.1°C). Tempered at 315°C for 10 sec. Etched with 4 pct nital and 1 pct zephiran chloride. Note completely austenitic rim. Bottom of picture shows martensitic plates formed toward the interior of the specimen. X100.

quenching. Fig. 7c shows how the core of transformation was changed in shape and shifted to the side of the specimen where the bending moment had added tensile components to counterbalance the residual compressive stresses in the rim.

Criterion for Effectiveness of Applied Elastic Stress

It has been demonstrated that a proper criterion for the initiation of the transformation by applied elastic stress must take into account the normal, as well as the shear, components. A feasible approach to the problem is derived from the nature of the transformation strains. The generation of a martensitic plate involves two displacements,[7] the first being a homogeneous strain that can be described as the motion of planes of no rotation and no distortion lying parallel to the habit plane. The direction of atom motion does not lie in this plane; hence the displacement is not one of simple shear. It may be assumed that this first strain is the primary displacement that occurs during the nucleation process. The second strain, which finally yields the martensitic structure, is homogeneous only over small regions compared to the volume of the plate, and will be regarded as playing no significant role in the activation phenomenon. The planes of no rotation and no distortion during the first strain slide past one another, corresponding to a shear strain of about 0.2; but they also move apart, corresponding to a normal (dilatation) strain of about 0.05.* Thus, the effectiveness of an applied stress should depend upon its ability to produce both shear and normal strains of the correct magnitude with respect to a potential habit orientation. Moreover, a normal compressive component will oppose the dilatation strain of the transformation, whereas a normal tensile component will aid it.† According to this concept the results of uniaxial tension and of uniaxial compression are expected to be quite different.

The foregoing combined strain criterion can be treated quantitatively through the use of a Mohr two-dimensional strain circle.[8] Because of the symmetry involved only plane strains need be considered and the strain circles are representative of any longitudinal plane passing through the tension or compression axis.‡ In this method of depicting plane strains, the principal strains (ϵ_1 and ϵ_3) are plotted along the x-axis from a common origin (Fig. 8) and a circle is constructed of radius $\frac{\epsilon_1 - \epsilon_3}{2}$, with ϵ_1 taken as the larger strain in the positive direction. The circle passes through ϵ_1 and ϵ_3 and has its center at $\frac{\epsilon_1 + \epsilon_3}{2}$. Letting ν = Poisson's ratio, then $\epsilon_2 = -\nu\epsilon_1$, where ϵ_1 is the principal (positive) strain parallel to the tension axis and ϵ_2 is the principal (negative) strain perpendicular to the tension

* These measurements were made on a 70 pct Fe–30 pct Ni alloy, having a {259} habit,[7] and are assumed to apply to the alloy under study here. Both alloys transform in approximately the same range of subzero temperatures and both exhibit burst phenomena.

† The sign of the shear component is of little consequence, since the many habit orientations virtually permit shearing in either direction.

‡ The plane strains are visualized to act on a potential habit plane whose normal lies in the plane of the strain circle. Thus the latter plane is perpendicular to the potential habit plane, and also contains the direction of atom motion during the first transformation strain.

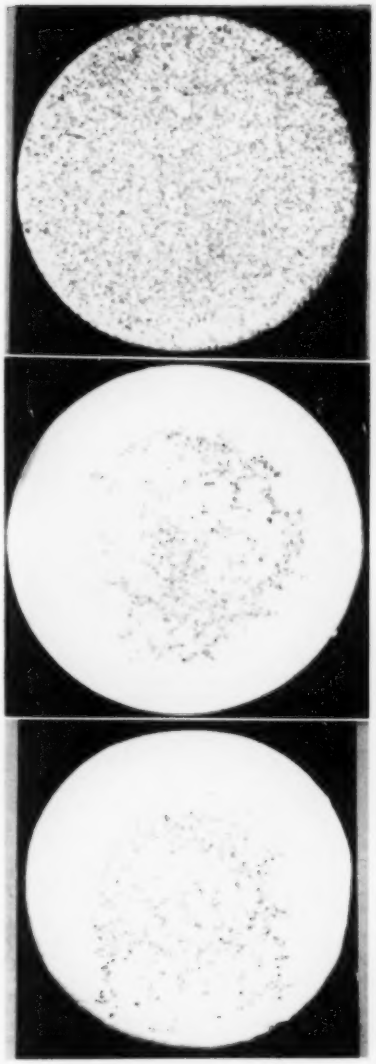


Fig. 7—Martensite formation in 20 pct Ni–0.5 pct C steel austenitized at 1095°C then treated as indicated. Finally tempered at 315°C for 10 sec and etched with 4 pct nital and 1 pct zephiran chloride. X10.

a—(top)—Oil quenched from austenitizing temperature, stress relieved at 480°C, and subcooled to M_s (—37°C). Note relatively uniform distribution of martensite across the transverse section.

b—(center)—Water quenched from austenitizing temperature and subcooled to M_s (—20°C). Note completely austenitic rim with transformation confined to the central portion of the section.

c—(bottom)—As in b, but with elastic bending moment superimposed on quenching stresses during the subcooling to M_s (—20°C). Note the shift of the transformed region toward the tension side (bottom of picture) of the applied bending moment.

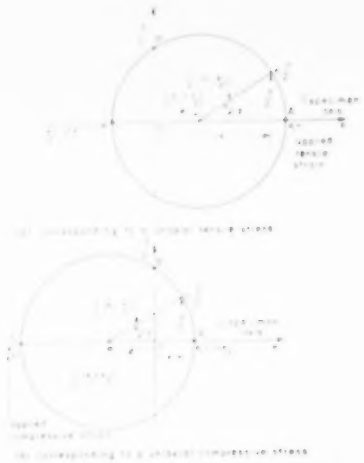


Fig. 8—Two dimensional Mohr strain circle depicting normal and shear strains as a function of orientation.

axis. Similarly for uniaxial compression, $\epsilon_1 = -\epsilon_2$, where ϵ_1 is the principal (negative) strain parallel to the compression axis, and ϵ_2 is the principal (positive) strain perpendicular to the compression axis. These two cases are illustrated in Fig. 8a and b.

A point on the strain circle lying at a radial angle 2α from the x-axis corresponds to a plane whose normal makes an angle α with the ϵ_1 direction. It can be shown that abscissa of such a point denotes the normal strain (ϵ) sustained by the plane in question and the ordinate of the point denotes one-half the shear strain $\left(\frac{\gamma}{2}\right)$ sustained by this plane. The mathematical relationship is:

$$\left(\epsilon - \frac{\epsilon_1 - \epsilon_2}{2}\right)^2 + \left(\frac{\gamma}{2}\right)^2 = \left(\epsilon_1 - \frac{\epsilon_1 + \epsilon_2}{2}\right)^2 \quad [1]$$

which is the equation of the strain circle. Because of the random orientation of the potential habit planes and the symmetry of the applied strain system about the specimen axis,[8] it is only necessary

[8] Also see the second (*) note on p. 665.

to consider orientations of α between 0 and 90°; this corresponds to points lying along the semicircle AHC in Fig. 8. Between A and B, ϵ and γ are positive and the orientations are such that transformation is aided by the acting strains. On the other hand, between B and C, ϵ is negative and these particular orientations sustain normal compressive strains that oppose the dilatation component of the transformation strain.

From a comparison of Fig. 8a and b, it is evident that arc AB (tension case) is greater than arc AB (compressive case), and hence there should be many more favorable orientations for transformation under uniaxial tension than under uniaxial compression. Yet even in the latter case, the surprising conclusion is reached that there should be some orientations that are aided by the uniaxial compression. (Actually, this results from the normal strains of opposite sign in the lateral direction due to Poisson's ratio.) However, since most of the orientations in this instance sustain normal compressive strains, a chain of reaction would tend to be inhibited, and M_s should be lowered even though M_d may be raised by the applied compressive stress. Thus, while $M_s = -37°C = M_d$ for the unstressed alloy, it is predicted that $M_s > -37°C > M_d$ for the uniaxially compressed alloy. Of course, under uniaxial tension both M_s and M_d should be raised according to the proposed criterion and this has been demonstrated already (Figs. 1 and 2).

To test the above prediction, austenitized and stress-relieved specimens were elastically loaded at room temperature, and electrical resistance changes were measured during subcooling with the specimens under constant stress. Uniaxial compression of 15,000 psi was applied in one run, and uniaxial tension of the same magnitude was applied in another. Unloaded specimens were also checked for comparative purposes. The resulting curves in Fig. 9 show that M_s and M_d were raised by 17°C in the tension experiment, while in the compression run, M_s was raised by 10°C and M_d was lowered by 6°C.

The relative effectiveness of uniaxial tension and compression in inducing the transformation can be calculated. Granting that the critical combined strain can be defined by a shear component γ_c and a normal component ϵ_c, eq 1 yields for uniaxial tension:

$$\epsilon_c \text{ (positive)} = \frac{-\epsilon_c(1-\nu) + \sqrt{\epsilon_c^2(1+\nu)^2 + \nu\gamma_c^2}}{2\nu} \quad [2]$$

and for uniaxial compression:

$$\epsilon_c \text{ (negative)} = \frac{-\epsilon_c(1-\nu) - \sqrt{\epsilon_c^2(1+\nu)^2 + \nu\gamma_c^2}}{2\nu} \quad [3]$$

It is then assumed that the critical components γ_c and ϵ_c stand in about the same ratio as the observed shear and normal components (0.2 and 0.05) of the

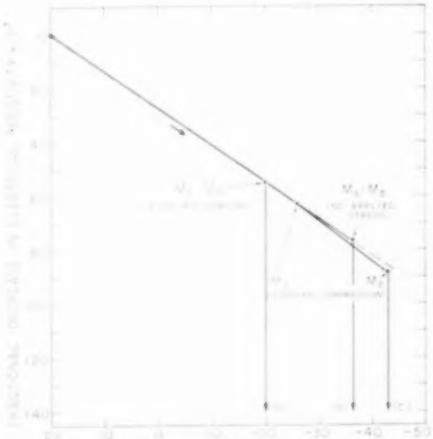
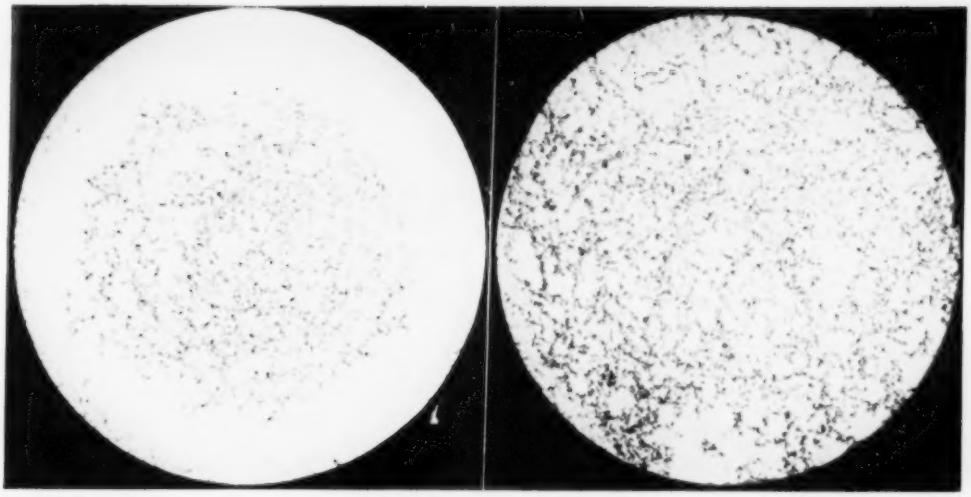


Fig. 9—Fractional decrease in electrical resistivity vs. temperature for specimen of 20 pct Ni 0.5 pct C steel cooled (a) without applied stress, (b) with uniaxial tensile stress of 15,000 psi, and (c) with uniaxial compressive stress of 15,000 psi.



a—Subcooled to −45°C with outer rim under triaxial compressive stress. Note lack of transformation there.

b—Recooled to −45°C without applied stress. Note relative uniformity of the transformation across the section.

Fig. 10—Martensite formation in 20 pct Ni-0.5 pct C steel austenitized at 1095°C, water quenched to room temperature, and subcooled as indicated. Finally tempered at 315°C for 10 sec and etched in 4 pct nital and 1 pct zephiran chloride. X10.

first transformation strain." Substituting in eqs 2 and 3 and letting $\nu = 0.3$, it follows that:

$$\epsilon_1 \text{ (compression)} = -1.8\,\epsilon_1 \text{ (tension)} \qquad [4]$$

This indicates that the uniaxial stress in compression required to initiate the transformation under a given set of conditions is 1.8 times the uniaxial stress in tension required to initiate the transformation under the same conditions.

A direct experiment to test this calculation has not yet been made, but the fact that the raising of M_s by 15,000 psi tension is 1.7 times the raising achieved by 15,000 psi compression suggests that the theory stands a good chance of being quantitatively correct. It would also give strong support to the reaction-path concept which provides the basis for the assumption that $\frac{\gamma_\epsilon}{\epsilon_1} = \frac{4}{1}$.

Experiments with Triaxial and Hydrostatic Compressive Stresses

The positive normal strains that exist in a uniaxially compressed specimen because of Poisson's ratio are eliminated in case of triaxial and hydrostatic compression. Furthermore, in the latter case, the shear components are also avoided. Therefore, the combined strain criterion indicates that both M_s and M_d should be lowered by triaxial and hydrostatic compression.

A 0.34 in. diam x 3 in. long specimen was austenitized and water quenched. Without being stress relieved, the specimen was copper plated and pressed into a central hole in an aluminum block, 2 in. OD x 1 in. long. The specimen was then loaded in uniaxial compression to an elastic stress of 15,000 psi and cooled slowly below room temperature. It was known from the experiments on residual stresses that the transformation would start in the core of the specimen due to the tensile stress there. This was found to occur as a burst at −31°C. (It is evident that the applied compressive stress partly cancelled the residual tension in the core and therefore the transformation set in at a lower temperature than if the compressive load had not been applied. Without such loading, the water-quenched specimen would start to transform in the core at about −26°C.) The aluminum has about the same thermal coefficient of contraction as the austenitic alloy, and hence it resisted the expansion due to the transformation in the core at −31°C. This generated compressive stresses transverse to the specimen axis (estimated to be comparable to the elastic limit) and the applied axial compression completed the state of triaxial compression. The lateral constraint provided by the aluminum block was definitely operative, as indicated by the fact that the applied axial stress jumped from 15,000 to 16,000 psi simultaneously with the burst. However, the applied load was quickly reduced to the original value in the testing machine before further cooling.

The specimen, triaxially compressed as described, was cooled to −45°C, held there for 5 min, returned to room temperature, and unloaded. The aluminum block was removed, and a portion of the specimen that had been surrounded by the block was tempered at 315°C to "darken" the martensite. From Fig. 10a, it can be seen that the core of the rod had undergone transformation, corresponding to the burst at −31°C, but the rim was austenitic even after the cooling to −45°C. The small amount of transformation visible near the edge of the specimen was undoubtedly an extraneous effect of the superficial distortion that occurred when the rod was forced into the aluminum block. When the same specimen was recooled to −45°C, under no external stress, the martensitic transformation took place in the pre-

visually untransformed rim, as shown in Fig. 10b. This experiment demonstrates that M_s and M_d can be depressed by triaxial compressive stresses.

Through the courtesy of Professor P. Bridgman at Harvard University, a few runs were carried out under hydrostatic pressure. With austenitized and stress-relieved specimens, the burst temperature was lowered from the customary −37°C to about −78°C by a pressure between 2000 and 4000 atm. The M_s temperature was also lowered by the hydrostatic pressure, but the extent was not determined with certainty. Further experiments along these lines are underway.

Summary

The formation of martensite in a 20 pct Ni-0.5 pct C steel under the application of stress has been studied as a function of the temperature of loading and the state of stress. The principal results may be summarized as follows:

1—The martensitic transformation can be initiated by elastic stresses at temperatures above M_s. Hence the role of applied stress is not merely one of producing nuclei by plastic deformation.

2—It is suggested that the role of applied stress at temperatures just above M_s is to supply critical strains to existing embryos. This makes it unnecessary to adopt the assumption that the austenite lattice becomes mechanically unstable at M_s.

3—The normal strain acting on a potential habit plane has an important effect on the nucleation process, and therefore a critical resolved shear stress or strain cannot be regarded as a valid criterion for inducing the transformation.

4—Shear strains and positive (tensile) normal strains acting on a potential habit plane promote the transformation, but negative (compressive) normal strains oppose it. Hence, strain energy, being independent of the sign of the normal strain, cannot provide a suitable criterion for the transformation.

5—Based on the reaction-path theory, a combined strain criterion is presented, which takes into account the shear and dilatation components of the atomic displacements during the transformation, and which is consistent with the following observations:

a—M_s and M_d are raised by uniaxial tension.

b—M_s is raised, but M_d is lowered, by uniaxial compression.

c—M_s and M_d are lowered by triaxial and hydrostatic compression.

6—Applied and residual stresses appear to be additive in their effect on the transformation.

7—Yielding by slip or by the stress-induced transformation may be considered as competitive deformation processes. Either may precede the other, and either may influence the other.

Acknowledgments

The authors wish to express their appreciation to Miriam E. Yoffa and to George Black of the Dept. of Metallurgy at the Massachusetts Institute of Technology for assistance in various phases of this investigation. The authors are also indebted to the Office of Naval Research for sponsorship of a research program of which this work was a part.

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aime NEWS

New England Regional Conference Delegates Hear Problems to Be Solved in Space Travel

A $4 BILLION research program would be sufficient to build and send aloft a space ship that would circle the earth, Dr. Fred L. Whipple, chairman, dept. of astronomy, Harvard University, told the 85 persons assembled at the banquet during the Sixth Annual New England Regional Conference, IMD, AIME, in Boston. This space ship, in addition to seeing and photographing what went on all over the world, would be an invaluable aid to meteorologists in forecasting weather, and could conduct a study of the sun. He said it would be necessary to fire this projectile at at least 5 miles per sec to get it away from the earth. With the known chemical fuels, it would be impossible to use a single stage rocket, and a multistage affair would be necessary. Dr. Whipple, who has done considerable work on rocket problems at White Sands, N. Mex. and similar projects, went on to relate various problems and possible solutions in designing, building, and equipping a space ship, and in sustaining human life aboard the craft. He also discussed problems to be encountered in space travel and methods for returning to earth, which are more difficult than the methods for leaving the earth. He mentioned that there would be little if any mining possibilities on the moon.

The banquet was formally opened by Al Blank, Chase Brass & Copper Co., chairman of the conference. He introduced as toastmaster E. A. (Andy) Anderson, New Jersey Zinc Co., who was chairman of IMD in 1947. Mr. Anderson spoke on behalf of AIME, pointed out the various Conference officials and guests seated at the head table, and then proceeded to introduce Dr. Whipple. The introduction of the Harvard man by a Yale man, and the following acknowledgment was enjoyed by the audience.

The Conference began Friday morning, April 25, with a plant trip to New England Metallurgical Corp., Boston, a large and well equipped heat treating plant. There the registrants were able to see at first hand flame and furnace brazing, armor plating, high speed salt bath treatments, cyaniding, tool and die heat treating, stress relieving and annealing, quench hardening and induction hardening.

The first technical session was held Friday afternoon. Associate chairmen for the session were D. K. Crampton, Chase Brass & Copper Co., and P. R. Kosting, Watertown Arsenal. M. C. Shaw, Massachusetts Institute of Technology, presented a paper on *Developments in the Theory of Metal Cutting*. He showed an excellent film on magnified views of cutting operations on various metals, and it could be seen readily that cutting is a shear effect. The influence of cutting fluids was clearly indicated.

J. H. Moore, National Research Corp., presented the early history, equipment, present and possible applications of vacuum equipment in a paper on *Vacuum Metallurgy and Its Applications*. He pointed out that while early vacuum casting experiments were limited to ingots of less than 300 lb, they were now casting 1800 lb ingots. *Problems in the Use of Copper and Its Alloys in the Electrical Industry* was discussed by W. A. Reich, General Electric Co. He pointed out the various alloys used and their applications, and went on to discuss substitution methods, and possible uses of aluminum. T. Y. Wilson, Allegheny Ludlum Steel Corp. gave a paper on *Continuous Casting of Steel*, bringing up to date the developments of the Rossi-Junghans continuous casting process.

The concluding session on metal failures was held Saturday morning under the chairmanship of A. J. Pepin, Wyman-Gordon Co. *Mechanism of Failure in Metals* was presented by Egon Orowan, Massachusetts Institute of Technology. He presented in a simplified manner the principle theories and formula determining metal failure. Charles Lipson, consultant, spoke on *Design Aspects of Metal Failures*. The principles of design, as well as specific design applications were discussed. M. H. Young, Wright Aeronautical Corp., concluded the meeting with a talk on *Fatigue Considerations in Aircraft Engine Development*. His talk centered on the design steps taken to prevent engine failures.

Registration for the Conference reached a record for the New England Regional Meetings of 180 AIME members and friends. The Conference Committee was composed of Albert I. Blank, Chase Brass & Copper Co., chairman; James H. Moore, National Research Corp., vice-chairman; Winston H. Sharp, Pratt & Whitney Aircraft, secretary; Leo P. Tarasov, Norton Co., treasurer; John A. Swift, Swift Industrial Chemical Co., past chairman; Michael B. Bever, Massachusetts Institute of Technology, chairman technical program committee; George S. Downing, General Electric Co.; Bruce S. Old, Arthur D. Little, Inc.; Lloyd E. Raymond, Singer Mfg. Co.; William L. Stearns, Bridgeport Brass Co.; Maur J. Weldon, Henry G. Thompson & Son Co.; and Franklin H. Wilson, American Brass Co.

Plans are already under way for the Seventh Annual New England Regional Meeting to be held under chairmanship of James H. Moore.

Final 1952 Budget Gains Board Approval

The final budget for 1952 was approved by the Board at its April 16 meeting. Income from initiation fees and dues is put at $326,000 ($306,000 in 1951); from publications $221,000 ($205,000 in 1951); other sources $7000 ($11,000 in 1951); total $554,000 ($522,000 last year). Expenses for membership services and field activities are budgeted at $107,000 ($81,000 in 1951); publications $311,000 ($294,000 in 1951); and general and administrative headquarters expense $129,000 ($139,000 last year).

Branchwise the budget was divided as follows: Mining income $265,500, expense $244,500. Metals income $163,400, expense $180,300. Petroleum income $125,100, expense $122,200.

An overall surplus for the year of $7000 is indicated.

Why AIME Members Should Vote For A Continuance of the Present Scale of Dues

By Michael L. Haider, AIME President

Three years ago AIME members were asked to vote on an increase of $5 in the annual dues for Members and Associate Members and of $2 for Junior Members. This increase in Institute income was made necessary by a long series of deficits, that for the year 1949 alone being $62,000. The increase was voted by 78.5 pct of the voting members of each of the Mining and Metals Branches and by 77.4 pct of the members of the Petroleum Branch. The proposal, later expressed in the bylaws, was that the increase should obtain for three years only, 1950, '51 and '52. It was felt that the situation should again be surveyed in 1952. You will therefore now again be called on to vote as to whether or not the present scale of dues shall be continued, as is recommended by the Board of Directors. You will receive a ballot during June if you are a paid-up member living in the United States, Canada or Mexico.

The increase in dues reduced the deficit in 1950 to $15,000, and last year a surplus of $3000 was achieved in spite of continuously rising costs. A surplus of about the same amount is indicated for 1952. Continuing surpluses are needed to replenish our emergency reserves depleted to meet past deficits.

The Institute income is largely derived from two sources, (1) Dues and initiation fees, and (2) advertising and sale of publications. Last year 59 pct of the income was from the first source and 39 pct from the second, leaving 2 pct from miscellaneous sources. In 1949 a new publication policy was adopted whereby we put out three magazines, each in specialized fields, instead of Mining and Metallurgy as previously. The objective of this policy was not only better to serve the members, but also to attempt to increase our advertising income. In 1949 advertising income was $80,000, compared with $103,000 in 1950, $144,000 in 1951, and $165,000 (estimated) in 1952.

Your Board has given serious consideration to reductions which might be made in Institute expenses. As a result of such studies, the Institute headquarters staff has been reorganized and equipment is being modernized. It is expected that increased efficiency and maximum operating economy will result. In spite of close scrutiny of costs, 1950 showed an increase of 7 pct over 1949, and 1951 increased 1 pct over 1950. Although the number of members increased each year, the number of regular paid staff employees was exactly the same at the end of 1951 as at the end of 1949.

A detailed analysis of the AIME income and expenses, both as to totals and on a per-member basis, covering the year 1951, appeared in the April issues of the Institute's three journals. Should the dues revert to their former level, it is estimated that income from this source would drop by approximately $69,000, or $3.50 per member including Student Associates. The resulting deficit would present a serious problem indeed as special funds which met the previous deficit have now been all but exhausted and your Board does not feel that drastic reduction in publications or staff are possible without serious impairment of services to members.

Most national engineering societies, and others in the mineral technology field, have been forced to increase their dues to offset inflation in printing and material costs as well as in salaries. In certain instances it has been possible for societies to escape the need for higher dues through having a comparatively large proportion of their income from sources other than dues, such as advertising, equipment shows, and profits on the sale of books.

The price level—the value of the dollar—is so uncertain in its present trend that income and expenses of the Institute are difficult to project into the future. If it should appear safe to do so, your Board of Directors has the power to reduce dues in the years ahead. On the other hand, dues can only be increased by vote of the membership.

You are strongly urged to return the ballot to be sent to North American members in June so that the returns will be as representative as possible of the wishes of the membership. You are also urged to give your Board of Directors a vote of confidence by authorizing them to continue the present scale of dues (Members and Associate Members, $20; Junior Members, $12 for the first six years of such membership, thereafter $17).

Annual Meeting Paper Deadline

Sept. 15, 1952 is the deadline for all Institute of Metals Div. papers for the 1953 Los Angeles Annual Meeting and the deadline for all Iron and Steel Div., and Extractive Metallurgy Div. papers to be prepublished.

MIT to Dedicate Metals Processing Lab

Massachusetts Institute of Technology's new Metals Processing Laboratory will be dedicated June 3, followed by a two-day technical session featuring leaders in the metal cutting field.

Alfred P. Sloan, Jr., chairman of the board of General Motors Corp., whose $1 million gift made the building possible, will speak at the dedication ceremonies. James R. Killian, Jr., president of MIT will deliver the acceptance speech.

A survey of the teaching and research activities in metal processing at MIT will be presented in the afternoon in two concurrent sessions: Mechanical Metallurgy, and Machine Tool Engineering and Metal Cutting. A series of laboratory visits to the new building will follow.

The conference on metal cutting, to be held June 4 and 5, is sponsored by the machine tool div., dept. of mechanical engineering. The conference will be divided in four sessions. Major themes of the sessions will be: Thermal aspects of High-Speed Machining, Wear and Tool Life, Applications of Metal Cutting Research to Machining Operations, and Mechanics of Cutting Process.

Student Conclave Attracts 128 Delegates to St. Louis

Some 128 students representing five universities attended the two-day student conclave held at Washington University, St. Louis, Mo., April 18. The opportunity for meeting industrial leaders on an informal basis was almost unanimously acclaimed as the outstanding feature of the event. Delegates came from St. Louis University, Missouri School of Mines and Metallurgy, Iowa State College, University of Illinois, and Washington University. L. P. Davidson opened the first day's session, telling how surplus funds from contributions made by various companies to the annual meeting, were used to finance the student conclave.

After the initial gathering, all delegates, St. Louis section officers, and faculty members had lunch, followed by four 20 min talks. A welcoming introduction by Dean Curtis L. Wilson, chairman of the St. Louis section started the proceedings. The students heard Dr. Frank C. Whitmore, Jr., of the U. S. Geological Survey speak on *Work in the USGA*; L. P. Davidson, general manager of American Zinc Co., of Illinois, on *What Can or Cannot a Metallurgist Be*; Noel H. Stearn, vice-president of the W. C. McBride Co., on the *Oil Industry*, and Francis Cameron, vice-president of the St. Joseph Lead Co., on *Where a Mining Company Offers an Opportunity and Career*. At the end, students and

speakers joined together for informal talks. The opportunity for talking freely with section officers and the representatives of industry continued during dinner that evening.

A car caravan formed by 30 students the next day traveled from St. Louis to Herculaneum. Enroute, students had the opportunity to study the geological formations of the area. Three St. Joseph Lead Co. men guided the students through the smelting plant. The caravan then moved to the Missouri School of Mines and Metallurgy at Rolla, joining those who did not make the trip. Two members of the U. S. Bureau of Mines addressed the students on *Hazards of Static Electricity*. Live demonstrations accompanied the lecture.

Dinner at the Edwin Long Hotel, Rolla, climaxed the conclave with Sparky Stalcup, Missouri University basketball coach, holding forth. Curtis L. Wilson expressed the section's gratitude for the reception given the idea of the conclave by the students. Paul Mudra, president of the St. Louis University Student Chapter expressed the thanks of the delegates.

The next Conclave will be held at Rolla in 1953.

Fullman Addresses Philadelphia Section

R. L. Fullman of the General Electric Co. addressed the meeting of the Philadelphia Section Apr. 17, 1952 at the University of Pennsylvania on *New Theories of Recrystallization and Grain Growth in Metals*.

Factors to be accounted for in any successful theory of recrystallization and grain growth were described by Fullman. He also discussed the variables which affect the rate of grain growth and rate of nucleation. Theories of oriented growth and oriented nucleation were compared. The speaker presented the results of his own experimental work as an answer to many of the questions that arose from the floor following the lecture.

Branch Council Heads Now Ex-Officio Directors

By vote of the Board at its April meeting, the bylaws were changed to make Branch Council Chairmen ex-officio Directors of the Institute. Division Chairmen have for some years been ex-officio Directors. Thus the total number of AIME Directors is now 39, consisting of 27 elected Directors, 10 Divisional Chairmen ex officio, and 2 Branch Chairmen ex officio. (The Petroleum Division Chairman is also the Petroleum Branch Council Chairman.)

Study of Hocott Proposal to Continue

At the April meeting of the AIME Board of Directors, E. E. Schumacher presented a report of the committee of which he was chairman that had been named to study the Hocott proposal. Main points in this proposal are that Local Sections be responsible to, and under the direction of, the Branch Councils rather than be administered by AIME headquarters; and that no more appropriations be made to Local Sections out of the AIME treasury—they would be financed by local dues and assessments. Four recommendations to the Board were made by the Schumacher committee: (1) There shall be no rebate of members' dues to Local Sections except as specified by individual members. (2) That the present Institute structure be continued. (3) That the authority of the Branches to serve the professional and administrative needs of local units be confirmed consistent with general Institute policy. (4) That a permanent Inter-Branch Council be established to study Institute organizational problems. This Council shall consist of (a) the Institute secretary, as permanent chairman; (b) the three Branch chairmen and immediate past chairmen or their designated alternates; and (c) the three Branch secretaries. This Council is to meet prior to the meeting of the Board of Directors at each Annual Meeting of the Institute and at any other necessary times.

The Board, at its April meeting on the 16th, voted to accept recommendations 2, 3, and 4, and to refer recommendation No. 1 to the committee authorized in recommendation No. 4 for future consideration.

No Change In Extractive Metallurgy Publications

At its April meeting, the Board again discussed the long-standing controversial problem of whether extractive metallurgy papers should be published in the JOURNAL OF METALS or MINING ENGINEERING, or both. Some members of the Extractive Metallurgy Div. prefer them to appear in one journal and some in the other. Strictly from the publishing standpoint, considering editorial coverage, circulation, and advertising income, the AIME is much better off to have such papers appear only in the JOURNAL OF METALS.

The Board decided to continue the present practice of publishing in MINING ENGINEERING only those extractive metallurgy papers so recommended by an auxiliary publications committee of the Mining Branch; to publish also brief abstracts in MINING ENGINEERING of extractive metallurgy papers that appear complete in JOURNAL OF METALS; and further to publish as much material in the JOURNAL OF METALS that is of interest to extractive metallurgists as is practical. Members receiving either journal may have a subscription to the other for an additional annual payment of $4.

Need Only Two Endorsers For Student Status Change

An appropriate change in Art. I, Sec. 9, of the bylaws was voted by the Board on April 16 whereby, in the case of Student Associates applying for change of status to Junior Member, only two endorsers are needed instead of three, provided one of them is the same member who endorsed his original application for Student Associateship.

Washington Award Goes To Henry Townley Heald

Henry Townley Heald received the Washington Award on April 21 "for distinguished leadership in engineering education, industrial technology, scientific research, and civic affairs." The AIME and other three Founder Societies, together with the Western Society of Engineers, recommend recipients for the Award through representatives on the Washington Award Commission. The Western Society of Engineers administers the award, which is an honor conferred upon an engineer for accomplishments which preeminently promote the happiness, comfort, and well-being of humanity.

Dr. Heald was recently made chancellor of New York University. Previous to that he was president of the Illinois Institute of Technology for 11 years and before that, at the age of 34, was made president of Armour Institute of Technology. He has served in one or more capacities in over 50 civic, social, religious, technical, and governmental groups.

AIME members who have received the Award in the past have included Herbert Hoover, Robert Woolston Hunt, Ambrose Swasey, F. G. Cottrell, D. C. Jackling, and Wilfred Sykes.

Members Urged to Return Cards For Directory List

As Section 2 of this issue is a directory of officers and committee members and AIME representatives on other committees. A complete AIME directory, similar to that issued in 1948 with the addition of the divisional enrollment of each member, will be issued later, probably in December. Members who have not returned the cards mailed to them for directory listing should do so immediately.

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Personals

Morse G. Dial has been elected president of Union Carbide & Carbon Corp. He joined the corporation in 1929 and became vice-president and director in 1949 and has been a member of the executive committee since 1951. Mr. Dial succeeds **Fred H. Haggerson**, who will continue as chairman of the board.

L. PAUL CLARE

L. Paul Clare is now metallurgical product engineer for the Sylvania Electric Products, Inc., tungsten and chemical div., Towanda, Pa. He had previously been associated with the Westinghouse Electric Corp., Buffalo.

Edward L. Aubuchon has resigned from the Wisconsin Steel Works and is now with the Austenal Laboratories, microcast div., Chicago, as metallurgical engineer.

William A. Casler, assistant director of research, has been named manager of program development at Armour Research Foundation, Chicago.

R. D. Bradford has been elected a vice-president of American Smelting & Refining Corp. **Richard G. Croft** was elected a director.

David L. Henry has joined the staff of Metal Hydrides, Inc. research laboratory as an assistant research chemist.

Robert M. Bracy is now an assistant metallurgist with Black Sivalls & Bryson, Kansas City, Mo.

Vern E. Dress has joined the Lockheed Aircraft Corp., Burbank, Calif., as process engineer.

Ward A. Wickwire, Jr. has been elected president of Salem-Brosius, Inc., Pittsburgh. Mr. Wickwire has been executive vice-president since 1951.

Arthur W. Lankenau resigned from the Denver Equipment Co. to accept a position with the National Lead Co., Fredericktown, Mo.

William C. Hunter has been made general foreman, reverberatory dept., smelter div., Phelps Dodge Corp., Ajo, Ariz.

John C. Murray, sales manager for the Brosius div., Salem-Brosius, Inc., Pittsburgh, and blast furnace consultant, Republic Steel Corp., is no longer connected with Salem-Brosius, Inc.

Vsevolod A. Gorsky has terminated his organizing work as manager of the Incorporated Gold Mines Ltd., Pietersburg, North Transvaal. He has resumed his mining and metallurgical consulting work in Johannesburg.

Arthur E. Hall is employed as consulting metallurgist for the Cia. Minera de Huehuetenango, Guatemala.

Robert T. C. Rasmussen is now chief, ferrous metals branch, metallurgical div., U. S. Bureau of Mines.

P. W. Ramsey is now with the A. O. Smith Corp., Milwaukee in the metallurgical research dept. He had been in the technical dept. of the N. J. Zinc Co.

Arthur R. Lytle has been appointed director of research. **Walter Crafts** has been named associate director of research, and **David Swan** was named assistant director of research, Union Carbide & Carbon Research Laboratories, Inc.

WALTER E. REMMERS

Walter E. Remmers has been elected vice-president, alloys div., Union Carbide & Carbon Corp. Mr. Remmers joined the firm in 1936 and has been president of Electro Metallurgical Co. since 1948 and president of U. S. Vanadium Co. since 1950.

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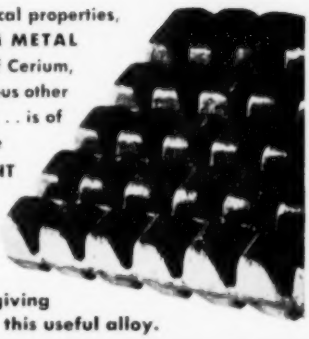

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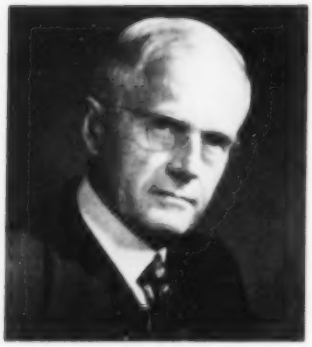

PAUL D. MERICA

Paul D. Merica, executive vice-president and a director, was elected president of the International Nickel Co. of Canada. **John F. Thompson** continues as chairman of the board and chief officer of the company.

Irving S. Olds, retired as chairman of the board of directors, U. S. Steel Corp. **Benjamin F. Fairless**, president, is his successor.

George M. Humphrey, president of the M. A. Hanna Co. for 23 years was elected board chairman and chief executive officer. **Joseph H. Thompson**, vice-president since 1937, was elected president and chief administrative officer. **George H. Love**, president of the Pittsburgh Consolidation Coal Co. was elected vice-chairman of the Hanna board and **R. L. Ireland**, a Hanna vice-president, was made a chairman of the executive committee.

Robert P. Parker was named director of chemical research, American Cyanamid Co., Calco chemical div., Bound Brook, N. J. **J. J. Denton** has been appointed assistant director of chemical research, pharmaceutical section.

Everett G. Couch, Jr. was recently appointed plant manager of the Kentucky works of the Chas. Taylor Sons Co., a subsidiary of National Lead Co.

Edwin H. Keiper has been made chief engineer of the Pennsylvania Crusher Co., Philadelphia. Mr. Keiper joined the company in 1919 and had been chief service engineer prior to his promotion.

James T. Towne has been appointed assistant to the vice-president, Vanadium Corp. of America, New York. He was formerly administrative assistant to the director of the research and engineering dept., Air Reduction Co.

Oscar G. Specht, Jr. is now senior technical and service representative, Electro Metallurgical Co., div. of Union Carbide & Carbon Corp.

Albert O. Trepuk is vice-president and treasurer of Wharton & Wilcocks of America, Inc., New York, a newly formed corporation. Mr. Trepuk is also president of the Douglas Export-Import Corp.

Harold R. Robinson is general mill superintendent, metallurgist, New York & Honduras Rosario Mining Co., San Juancito, Honduras.

William A. Arpi has been promoted to assistant superintendent of the milling dept., Tennessee Copper Co., Isabella, Tenn. He had been metallurgical engineer.

Weston Bourret, formerly geologist with Kennecott Copper Corp., New York is now chief, minor and light metals branch, Defense Materials Procurement Administration, Washington, D. C.

Alexander Cochardt is research engineer with the Westinghouse Research Laboratories, East Pittsburgh.

Grenville H. Grimwood has joined the British Metal Corp. (Canada) Ltd., Vancouver. He had been chief metallurgist for the Canadian Explorations Ltd.

Lawrence H. Van Vlack is now process metallurgist with the U. S. Steel Co., Pittsburgh.

D. L. McBride has become staff metallurgist for the National Academy of Sciences, minerals and metals advisory board, Washington, D. C.

Obituaries

Robert C. Coffey (Member 1922) died during January 1952. Born in Kansas on Feb. 1, 1883, Mr. Coffey attended Iowa State College, graduating in 1905. Following graduation he worked in Iowa, Colorado, and Nevada as surveyor, cyanide foreman, and mill foreman. In 1910 Mr. Coffey was cyanide superintendent and mill superintendent for the Star Mine. He then joined the St. Anthony Gold Mine at Sturgeon Lake, Ont., and later became mill superintendent for the Dominion Reduction Co., Cobalt, Ont. Mr. Coffey went to North Carolina in 1913 as mill superintendent for the Uwarri Mining Co. He returned to Ontario as mill superintendent for the Schumacher Gold Mines, Ltd. In 1916 he was engaged in work on the erection of the cyanide plant for Lake Shore Mines, Ltd., and in 1918 was made manager. Several years were spent as consulting engineer for the Ontario Kirkland Gold Mines Ltd., and in 1912 he was made president of the Kirkland Lake Mine Managers Assn. At the time of his death, Mr. Coffey was residing at Orlando, Fla.

Proposed for Membership
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Total *AIME* membership on Mar. 31, 1952 was 17,312; in addition 2260 Student Associates were enrolled.

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The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

In the following, an (S) means change of status. R, reinstatement; M, Member; J, Junior Member; A, Associate Member; S, Student Associate.

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NECROLOGY

Date Elected	Name	Date of Death
1905	Frank F. Colcord	Mar. 21, 1952
1935	E. A. Collins	Feb. 6, 1952
1939	Charles H. Davis	June 1951
1905	Rolls F. Edwards	Aug. 6, 1951
1939	William McK. Green	Mar. 12, 1952
1896	John Gross	Apr. 2, 1952
1951	Thomas M. Harris	Unknown
1944	George H. Jones	Unknown
1939	H. M. Lavender	Mar. 21, 1952
1928	Charles Ellison MacQuigg	Apr. 24, 1952
1897	Eugene L. Messler	Unknown
1916	Rossi B. Rathbun	Apr. 3, 1952
1895	A. H. P. Wynne	Unknown

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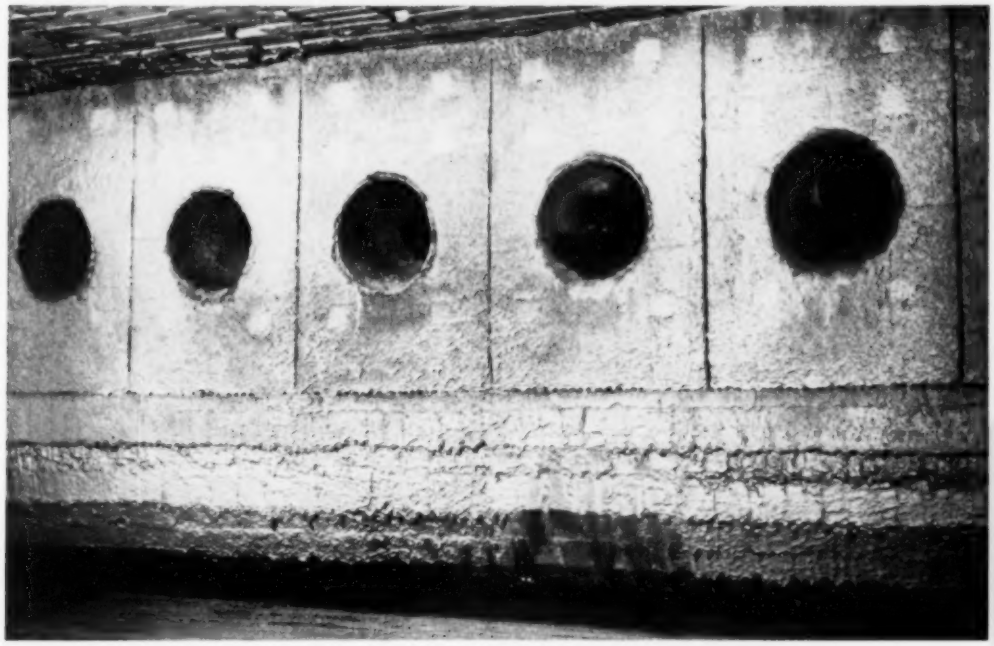

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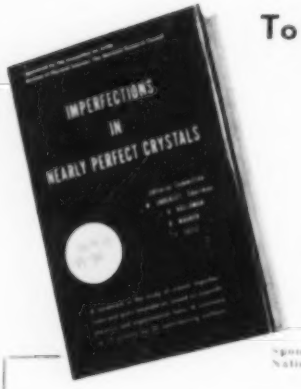

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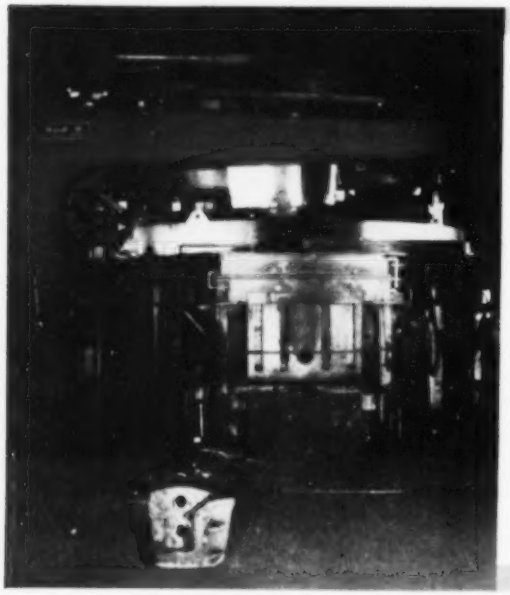

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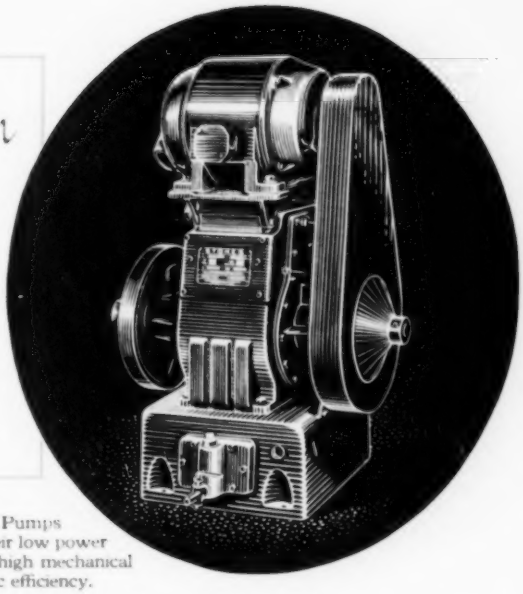

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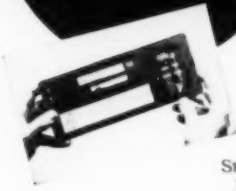

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JUNE 1952, VOL. 4, No. 6

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OF

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CORRECTED TO MAY 1, 1952

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Upper Peninsula—Roy W. Drier
Utah—Whitman G. Rouillard
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Established November 19, 1947
Regular meetings fourth Monday of each month except June, July, and August

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Robert M. Chapman, Vice-Chairman
Robert E. Shafer, Secretary-Treasurer
Box 1170
Fairbanks, Alaska
Robert M. Chapman, Section Delegate

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Established July 10, 1915

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Established December 13, 1936

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Established September 20, 1911
Meets First Monday of each month
October to May inclusive

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Massachusetts Institute of Technology
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Established September 19, 1946
Meets third Thursday of each month
except July and August

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H. P. Clark, Second Vice-Chairman
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Established October 17, 1940

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Established February 26, 1914
Holds dinner meeting first Wednesday
of each month, October to May inclusive,
at the Chicago Bar Association

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Inland Steel Co.
East Chicago, Indiana
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Established April 17, 1936

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Established May 23, 1913

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Cripple Creek, Colorado

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Established November 24, 1911
Holds meetings first Friday of each month except July and August

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D. L. Myers, Secretary-Treasurer
3827 W. Kiernan
Spokane, Washington
Lewis A. Grant, Section Delegate

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Northwest Industrial Minerals and Metals Branch Conference
Spokane, May 1952
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Established September 30, 1947

Holds meetings second Wednesday, October, December, February and April

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William Mounce, Membership
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Established September 18, 1946

Holds meetings second Tuesday each month except June, July and August

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Established February 18, 1936

Holds meetings third Monday of each month except June through September

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Established April 18, 1940

Holds meetings second Tuesday of each month except July through September

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Established March 25, 1927

Holds meetings second Wednesday of each month

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912 W. Yandell Blvd.
El Paso, Texas

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March—J. C. Rintelen, Jr.
April—Ben D. Roberts
May—Mallory L. Miller
September—A. L. Washburn
October—Randolph S. Murray, Jr.
November—Eugene M. Thomas
December—Paul F. Deisler
January—Russell R. Whittington

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PUBLICITY

H. F. Kannady, Chairman

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Established September 27, 1949

Holds meetings the first Monday of each month

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Walter O. McClintock, Secretary-Treasurer
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Bartow, Florida
Allen T. Cole, Section Delegate
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Ernest Greene, Steering
John Yost, Membership
Lawrence Frye, Program

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Established December 13, 1935

Holds meetings the third Tuesday of each month

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R. M. Rutledge, Secretary-Treasurer
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P. O. Box 1407
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B. C. Elsley
W. D. Smith, Jr.
E. F. Stratton
Walter Rose
C. G. Wrightsman

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Established November 17, 1948

Holds meetings third Wednesday of each month except June through August

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G. J. Decker, Vice-Chairman (*Wichita*)
E. S. Arnold, Vice-Chairman (*E. Kansas*)
J.W. Meyers, Vice-Chairman (*W. Kansas*)
H. L. Temple, Secretary-Treasurer
Continental Oil Co.
Moundridge, Kansas
G. L. Yates, Section Delegate

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B. B. Lane — Lyman Terry

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L. L. Terry — R. V. Gerner

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Will D. Ford — John T. Gary
Dennis Krehbiel — Mac. W. Miller

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Established March 27, 1924

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J. S. Marsh, Vice-Chairman
J. L. Rodda, Vice-Chairman
D. J. Blickwede, Secretary
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Bethlehem, Pa.
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R. T. Gallagher, Section Delegate
B. J. Larpenteur, Manager
W. A. Griffith, Manager
R. T. Gallagher, Manager

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J. P. Bingham — R. W. Jenkins
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Allison Butts — C. E. Taylor, Jr.
M. S. Childs — P. L. Steffensen

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Established December 28, 1917

Holds weekly luncheon meetings each Thursday in Tulsa, Oklahoma. Regular Section meetings held second Monday each month except June through August.

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C. C. MacDonald, First Vice-Chairman
M. B. Penn, Second Vice-Chairman
S. F. Bird, Secretary-Treasurer
Amerada Petroleum Corporation
Tulsa, Oklahoma
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E. H. Nielsen
F. H. Calloway

(OUTSIDE TULSA)
J. A. Murphy
F. H. Poettman

(EX-OFFICIO)
Roy H. Smith
J. N. McGirl

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Established December 20, 1920

Annual meeting second week in January, other meetings as called by officers

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J. L. Severson, Vice-Chairman
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Box 840, Hibbing, Minn.
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M. F. Williams, Secretary

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Established October 17, 1913

Holds meetings fourth Tuesday of each month

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Clifford J. Hicks, Secretary-Treasurer
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Joseph T. Roy, Executive Committee
James F. Smith, Executive Committee
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Stanley M. Lane — Forbes Robertson
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Holds luncheon meeting second Friday each month except July and August

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Established May 19, 1911

Holds monthly meetings except February, June, July, and August

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Established March 28, 1913

Holds meetings third Thursday each month except June, July, and August.

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Established February 17, 1914

Holds meetings alternately at Pottsville, Hazleton, Wilkes-Barre, and Scranton. Spring Meeting, April; Summer Meeting, July; Fall Meeting, October.

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Established June 26, 1913

Holds meetings second Wednesday of each month except June through August; and Monday noons for luncheon at Engineers' Club

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Established September 13, 1946

Holds meetings second Wednesday of each month except July, August and December

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Established February 1951

Holds meetings fourth Monday of each month.

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Established September 30, 1947

Holds meetings second Wednesday in January, April, July, and October.

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Established April 24, 1914

Meets third Thursday of each month, September to May, inclusive

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Established June 21, 1918
Holds meetings usually third Tuesday of each month except June through August.

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Established May 16, 1930

Holds regular meetings quarterly; special meetings when speakers are available. (Officers' terms expire Nov. 1953)

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Holds monthly luncheon meetings first Monday of each month at American Club, Mexico City

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Established April 17, 1936

Meets second Thursday of each month at Av. Rio Branco 128, Room 410

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